THE MANUFACTURE OF
EXPLOSIVES

TWENTY YEARS' PROGRESS

FOUR CANTOR LECTURES DELIVERED AT THE ROYAL SOCIETY OF ARTS IN NOVEMBER AND DECEMBER 1908

BY

OSCAR GUTTMANN

MEMBER OF THE INSTITUTION OF CIVIL ENGINEERS; FELLOW AND VICE-PRESIDENT OF THE INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND, FELLOW OF THE CHEMICAL SOCIETY, MEMBER OF THE INSTITUTION OF MINING AND METALLURGY, MEMBER OF THE SOCIETIES OF CIVIL ENGINEERS AND ARCHITECTS OF AUSTRIA AND HUNGARY, CORRESPONDING MEMBER OF THE IMP. ROY GEOLOGICAL INSTITUTION OF AUSTRIA, ETC

WITH ELEVEN ILLUSTRATIONS AND COLOURED FRONTISPIECE

WHITTAKER AND CO.

2 WHITE HART STREET, PATERNOSTER SQUARE, LONDON, E.C.

AND 64 & 66 FIFTH AVENUE, NEW YORK

1909

1450

TTMANN.

ufacture

ives.

RICHARD CLAY & SONS, LIMITED,
BREAD STREET HILL, E.C., AND
BUNGAY, SUFFOLK.

PREFACE

THE series of Cantor Lectures forming the subject of the present volume were written at the suggestion of my friend, Sir Boverton Redwood, and delivered before the Royal Society of Arts on the 23rd and 30th November, and 7th and 14th December, 1908, with whose permission they are reprinted.

In so small a compass it was only possible to give a general outline of the many improvements and researches effected during the last twenty years and a summary of the most important results achieved. I have, however, endeavoured to make these lectures of real value by discoursing on results which are not generally known, and by critical consideration of important processes and problems

This requires a certain amount of courage, since it is sometimes impossible to avoid clashing with the views, business interests, or even the vanity of individuals. It will, however, be acknowledged that in all my literary work I have endeavoured to be scrupulously impartial.

These lectures required not only many months of preparation, but also most comprehensive inquiry and research. This was only rendered possible by the extensive assistance of friends, authorities, and colleagues, and I therefore tender them herewith my heartfelt gratitude.

OSCAR GUTTMANN.

London, January 1, 1909

CONTENTS

LECTURE I.

State of the Art in 1886—Black powder—Other nitrate mixtures—Perchlorat in saltpetre—Machinery—Chlorate mixtures—"Metallic" explosives—Picric acid and picrates—Trinitrotoluol . . . p. 1 to 1

LECTURE II.

Manufacture of nitroglycerine—Use of anhydrous sulphuric acid—Displacement process—Abolition of after-separation—Dynamites—Unfreezabl nitroglycerine explosives—Guncotton—Selection of cotton—Acid mixtures—Apparatus—Percentage of nitrogen—After-treatment—Nitrostarc p. 17 to 3

LECTURE III.

History of smokeless powders—Their manufacture—Flameless powders—Fulminates and detonators—Fuses—Safety explosives—What renders a explosive safe in fire-damp?—When is an explosive safe?—Coal dust—Safety explosives in practice . . p. 36 to 5

LECTURE IV.

Use of nitrocellulose for celluloid, artificial silk and varnish—Machiner driven by explosives—Factories, their inspection, construction and accidents—Precautions recommended—Electric lighting—Static electri charges—The merits and demerits of explosives—Stability and its proof—Stability in practice—Accidents with smokeless powder—Stabilizers—The powder of the future . . . . p. 54 to 6

APPENDICES . . . . . . p. 71 to 8

INDEX . . . . p 81 to 8

LIST OF ILLUSTRATIONS

FIG		To face page
	Ordinary and Nitrated Cotton seen under the Microscope in Polarized Light	*Frontispiece*
1.	Nitrator Separator (Nathan, Thomson and Rintoul's Patent)	21
2.	Calcined Kieselguhr	22
3	Nitrating Centrifugals	32
4	Nitration of Cotton (Thomson's Patent Displacement Process)	34
5	Smokeless Bulk Powder seen under the Microscope in Ordinary Light	44
6.	Smokeless Bulk Powder seen under the Microscope in Polarized Light	44
7.	Smokeless Flake Powder seen under the Microscope in Ordinary Light	44
8.	Smokeless Flake Powder seen under the Microscope in Polarized Light	44
9.	Testing Station at Frameries (End View)	50
10.	Testing Station at Frameries (View of the Interior)	50
11	Explosives Workshop and Mound Built in Ferro-concrete	56

TWENTY YEARS' PROGRESS IN EXPLOSIVES

I

About twenty-five years ago this Society showed its interest in Explosives by offering a prize for a process or device calculated to minimize danger during manufacture. Although I once gave a lecture on these dangers I have not come to claim the prize; thirty-two years of intimate work with explosives have taught me to be modest about inventions. I have, however, endeavoured from time to time to keep the world in touch with this industry, and am grateful to the Council of this Society, which has exercised such a far-reaching influence on British industry, for having invited me to review the progress of the last twenty years.

I doubt whether there has been a more important period in the history of explosives than the last twenty years, or one in which more improvements of real value have been brought forward. A short historical retrospect will be very useful in order to fully appreciate this fact. From the time of the invention of gunpowder, or approximately in the year 1250 (Roger Bacon at any rate knew of it in 1264), until the beginning of the nineteenth century, no other explosive was introduced into practice, although picric acid and fulminate of mercury were known about the latter date. Experiments were carried out by Le Blond in 1756 in the French Government factory at Essonne to produce gunpowder without sulphur, and a British patent for a powder containing "coal brasses" and without charcoal was taken out by Delaval in 1766,[1] but that was all. In 1788 Berthollet and Lavoisier tried the effect of adding potassium chlorate, and in 1861 Designolle made a powder from potassium picrate and saltpetre, but without

[1] Thomas Delaval, British patent, No. 846 of 1766.

much success. In 1846 Schoenbein invented gun-cotton, and Sobrero in 1847 nitroglycerine, but the Austrian Government, which was the only one to try gun-cotton in guns, stopped the experiments abruptly in 1867, their magazines at Hirtenberg having blown up, and, curiously enough, it is not until that date that Nobel begins to work on dynamite About the same time the British Government began to experiment with gun-cotton at the point where the Austrians had left off, and introduced it as a blasting agent into the Service, their example being then followed by other Governments. In 1873 Sprengel made his well-known communication to the Chemical Society "on a new class of explosives," which has since been named after him; and in 1878 it was again Nobel who invented blasting gelatine. About 1864 Abel and Dr. Kellner, of Woolwich Arsenal, made a granular gunpowder from gun-cotton, and at the same time a sporting powder from nitrated wood, the Schultze powder, was introduced. Later on, in 1882, Reid made grains of soluble gun-cotton, and hardened them by means of ether-alcohol, calling the product E. C. powder. In the third lecture reference will be made to the important smokeless powder of Friedrich Volkmann made in 1870.

Such was the state of the art in 1886, when simultaneously Eugène Turpin, of Paris, suggested the use of compressed or molten picric acid as a charge for shells, and Vieille carried out his famous experiments, resulting in the manufacture of the Poudre B (so named after General Boulanger). At the same time it was recognized, that most explosions in coal mines were due to the ignition of fire-damp by the firing of shots, and that it was possible to make so-called "safety explosives" which would considerably reduce this danger.

Hereafter investigations and inventions came in almost too rapid succession. Unfreezable dynamites, dinitroglycerine explosives, picric acid compounds and trinitrotoluene explosives, fulminates from aromatic nitro-compounds, phlegmatized fulminate, detonating fuses, and many other varieties were invented Nitrocellulose, than which there is hardly a more complex substance, was investigated by Cross and Bevan, Haussermann, Lunge, de Mosenthal, Vignon, Will, and others; the stability of nitro-compounds, the properties of nitroglycerine, and many other substances investigated by an army of workers. In fact, quite as important results have been obtained since 1886 as in the whole of the previous years. This is due in the first instance to the enormous amount of scientific research and experiment devoted by

manufacturers to the study of such questions, partly because they were forced to do so by considerations of national defence, the advent of the rock-drill, and by competition, and partly because those who lacked the training for such research could be persuaded by the results achieved to appreciate the work of others. Whilst until a generation ago the so-called powder-maker was a craftsman, who carefully guarded little *tours-de-main* as valuable trade secrets, and even the inventors of high explosives had to advance in a most empirical way, it is recognized now-a-days that only the best scientific knowledge can effect improvements or keep in line with modern developments of the industry. I have stated on another occasion[1] how large an amount of engineering and manufacturing knowledge is required for high explosives factories, since these generally make their own prime materials, work up their waste products, and are therefore chemical works to a much greater extent than explosives works, whilst at the same time the fact that they cover large areas of ground necessitates the introduction of very special arrangements.

In common with others I once thought that the use of black powder was dying out, and perhaps this will actually be the case one day; but I confess that I did not reckon sufficiently with the conservative inclination of the miner, who in very many cases clings most tenaciously to the old black powder. Whilst for warlike purposes the use of black powder, and even that of the later brown powder, has become a negligible quantity, blasting powder is still sold to such an extent, that in the mines and quarries of this country alone nearly 7000 tons of it, or more than half the total weight of all explosives, were used in 1907[2] This of course represents only part of the total quantity manufactured in this country, since 3597 tons of gunpowder of all kinds of British and Irish production were exported,[3] and a good deal was used for railways, roads, etc.

There has been practically no progress made in black powder within the last twenty years Brown powder, which, as is known, contained slack burnt charcoal and a small percentage of sulphur, greatly improved the shooting of large guns, but has gradually given way to smokeless powder, even for the very largest guns. A little black powder is still used as a primer for large charges, but

[1] "Minutes of Proceedings of the Institution of Civil Engineers," 1901, vol. cxlii, part i.

[2] "Report of H.M. Inspectors of Explosives for 1907."

[3] Private communication.

even for that purpose it will gradually be replaced by specially prepared smokeless powder. There are still some old sportsmen who prefer to use nothing but the old fine black sporting powder, and this is more especially the case in remote parts of Germany, Austria, and Italy, whilst in the United States of America professional sportsmen, *i.e.* those who shoot wildfowl for the market, use black powder because of its cheapness. There is a certain amount of competition going on in this quarter with smokeless powder, and manufacturers of black sporting powder are thereby obliged to make special efforts to produce material of the highest grade only. This has revived interest to some extent in the methods of manufacture of black powder. It is curious to find that the so-called improvements in this manufacture are really revivals of old methods; thus, for instance, mixing powder in drums has in some instances replaced the use of incorporating mills, and the so-called Swiss or "Nassbrand" (wet residue) powder, which is a powder consisting of perfectly globular grains of uniform size, and of such composition as to give a residue absorbing moisture quickly, has again found some favour. The machinery for rounding this powder is still the same as that described one hundred and forty years ago.[1]

The enormous development of the German potash industry, and the peculiar requirements of potash and salt mining, have also revived some rough mixtures of black-powder-like explosives, of which very large quantities are now sold in Germany. It will be remembered that already in 1865 G. A. Neumayer tested a special powder at the Stassfurt salt mines.[2] Now-a-days "Sprengsalpeter" is made, consisting of 75 per cent. of sodium nitrate, 15 per cent. of brown coal, and 10 per cent. of sulphur, or practically a black powder with sodium nitrate instead of potassium nitrate. Another explosive of this kind is "Petroklastite,"[3] containing coal-pitch and bichromate. Yet another such explosive is "Cahuecite," which is our old friend "Safety blasting powder," made at Dartford twenty-five years ago. It contained potassium nitrate, sulphur, lampblack, cellulose, and iron sulphate, and is made by boiling the mixture in steam-heated pans. All these powders burn slowly when ignited, and fissure the rock rather than shatter it.

In America, also, large quantities of black powder made with sodium nitrate are used. Labour there is so expensive that

[1] "Recueil de planches sur les sciences, les arts libéraux et les arts méchaniques avec leur explication" Paris, 1768.

[2] "Dingler's Polytechnisches Journal," 1866, p 248 and foll

[3] F. Heise, "Sprengstoffe und Zündung der Sprengschüsse," Berlin, 1904, p. 46.

work is done with this cheap explosive which on this side of the Atlantic would be carried out with pick and shovel.

Progress of a different kind has been effected by using ammonium nitrate as an ingredient in a powder mixture. This also was tried in France in the eighteenth century with but little result.[1] Amide powder,[2] however, made by the Koeln-Rottweil Works, and consisting of 40 parts of potassium nitrate, 38 parts of ammonium nitrate, and 22 parts of charcoal, might, but for the advent of smokeless powder, have become a serious rival to black powder. Mayer, of Felixdorf, in Austria, also worked in this direction. The Austrian Government makes Wetter-Dynammon as an explosive for fiery mines, which, according to Ulzer,[3] consists of 93 83 per cent of ammonium nitrate, 1·98 per cent. potassium nitrate, 3 77 per cent charcoal, and 0 42 per cent. moisture, the charcoal grains being 1 to 6 μ in size.

Further progress, although seemingly small, has been that made in powder for safety fuses. As is known, safety fuses can be compared to a piece of cord, having a fine train of gunpowder in the centre. Competition has, in this case also, given the consumer the opportunity of demanding a safer and more reliable article than before. It is most important that the fuse should burn at a definite rate per second, and that the powder should not explode in certain parts of the fuse and hang fire or cease to burn in others. Many accidents have occurred in mines through bad fuses delaying the shot, or smouldering for hours. Formerly it was not uncommon to use the siftings from mining powder for safety fuses, but the present stringent requirements have compelled all manufacturers to make a special quality of fuse powder of constant composition, density, and uniformity of granulation, in spite of its almost dust-like character.

I shall lay before you later on some information concerning safety explosives for fiery mines, and, therefore, will only mention that in every European country the use of gunpowder is prohibited in such workings. Considerable surprise was therefore felt when several black-powder-like mixtures passed the official test for permitted explosives in this country. Later, when these tests were made more rigorous, these explosives disappeared, but one of them, Bobbinite, passed even the more stringent tests,

[1] Bottée et Riffault, "Traité de l'art de fabriquer la poudre à canon." Paris, 1811

[2] Gaens, British patent, No 14412 of 1885.

[3] "Mitteilungen des tech nologischen Gewerbemuseums," Wien, 1900, p. 204

and is still on the new list of permitted explosives. According to the official definition it consists of about 64 parts of potassium nitrate, 2 parts of sulphur, and 19 parts of charcoal, with the addition of 15 parts of a mixture of ammonium- and copper-sulphates, or alternatively of 8 parts of starch and 3 parts of paraffin wax, with a corresponding increase in the other materials. We will examine, later on, the reasons why such a powder mixture does not ignite fire-damp, whilst ordinary powder does.

Complaints having been made as to the alleged danger of Bobbinite in fiery mines, the Home Office appointed a departmental committee in 1906 to investigate the matter, which came to the conclusion that the use of Bobbinite should not for the present be restricted.[1] The importance of this explosive may be gauged from the fact that over a million pounds of Bobbinite were used in 1907 in this country. A slow-burning explosive, which does not rend the coal too much, has its advantages, and the fact that the miners are used to black powder, and that if the borehole be overcharged as usual, Bobbinite does not break up the coal badly, has also assisted in making it popular.

Quite a scare was produced in 1894 by the discovery, made by Major Hellich, that so-called "German saltpetre" contained perchlorate as well as chlorate,[2] and that Dr. Panaotovic, of the Royal Servian Gunpowder Factory of Stragare, was able to prove by the re-examination of old samples, that most of the accidents in his factory coincided with a considerable percentage of perchlorate found in the powder, which caused the explosion.[3] Many investigations followed, evidence of accidents seemed to accumulate, the German quarry owners desired to invoke legislation, manufacturers tried to find means to eliminate the perchlorate, and as usual a number of investigators tried to find the best method for proper analysis. We did not take such a serious view of it in this country, but when the alarming foreign statements could not be overlooked, Mr Bertram Blount and myself considered the case for certain regular large deliveries, and we came to the conclusion that 0·1 per cent. of perchlorate could be fairly fixed as a maximum for the makers of saltpetre, whilst such a small quantity would be so finely distributed in a well-made powder as to cause no danger when the usual methods of charging a borehole are observed. All this, however, became

[1] "Report of the Departmental Committee on Bobbinite." London, 1907.
[2] "Chemiker Zeitung," 1894, p. 485
[3] *Ibid.*, p. 1567.

superfluous in the end, since Dr. Dupré in this country,[1] and Professors Lenze and Bergmann in Germany,[2] showed conclusively that, even with extraordinarily rough treatment, powder containing perchlorate was no more dangerous than one without it. At the same time Dr. Dupré found, that by heating saltpetre to a temperature of 545° C., the whole of the perchlorate was converted into chlorate, and therefore could be got rid of in extreme cases.

Kelbetz[3] had shown that perchlorate is not uniformly distributed in saltpetre, but tends to agglomerate. There can be no doubt that perchlorate is more explosive than the powder itself, and it therefore occurred to me that this might be the reason why certain English black sporting powders are preferred to foreign ones, which are supposed to be less regular in shooting. These English powders are made with Indian saltpetre, which does not contain any perchlorate, whilst the foreign powders are made with potassium nitrate, which is manufactured by conversion from the Strassfurt potash salts, and always contains perchlorate. Undoubtedly strong local action will cause a powder to behave somewhat erratically. At the Congress for Applied Chemistry held in Berlin in 1903, Professor Bergmann took special trouble to refute my contention, but two plain facts could not be controverted, namely, that even German sportsmen found certain English sporting powders to be superior, and that when using "German saltpetre" this could not be altered even by the most careful and strict observation of the usual manufacturing routine.

With regard to machinery used in the manufacture of black powder and similar mixtures there has, of course, been very little improvement. Mixing, granulating, and glazing are still carried out in the same way, and for the purpose which they have to accomplish the machines do all that can be desired A good deal of ebonite was formerly used in connection with machinery for black powder, such as for plates in cake presses, for lining the hoppers of cutting machines, etc. In cake presses there are alternate layers of powder containing sulphur, and of highly insulating ebonite, which remain together under pressure for some time. It is a rule in explosives works that at the approach of a thunderstorm the workers leave their houses, and it is frequently found convenient, meanwhile, to leave the charge under pressure.

[1] "Journal of the Society of Chemical Industry," 1902, p 825

[2] "Ueber Perchlorat im Schwarzpulver und über Gefahren bei der Fabrikation und Verwendung perchlorathaltiger Schwarzpulver" Paper read before the V International Congress of Applied Chemistry in Berlin, 1903.

[3] "Chemiker Zeitung," 1897, p. 587.

This would practically constitute an electric pile, and as a matter of fact several explosions have occurred when, after the thunderstorm, the workers opened the presses In one instance, at least, the fact of a long spark having come out of the charge could be elicited from the attendant before his death.

Following a suggestion made by the author twenty years ago, a number of factories have substituted plates of fibre for these ebonite plates with great success Fibre is a material made of paper pulp with the addition of certain mineral matter and binding material. It has the advantage of not becoming charged like ebonite, of not warping easily, and when warped of becoming flat again by immersion in tepid water, which treatment at the same time softens the material should it have become hard

Black powder is still sifted in some factories in the old-fashioned frames hung on springs, but more frequently this is now performed in cylinders. In both cases a good deal of dust is produced, which affects the quality of the powder by closing its pores, whilst it also has to be removed as so much waste. One of the improvements effected is the use of a plane sifter. It imparts a slight motion of high frequency, and thus the grains are not subjected to violent friction or impact.

Chlorate mixtures have at all times fascinated inventors on account of the large amount of oxygen stored up in potassium chlorate, which can be given off so readily. When Lavoisier and Berthollet tried to make a chlorate powder in a stamp mill in 1788, they made a great show of it, and even two ladies were present. Unfortunately after a certain amount of pounding the powder exploded and killed an official and the daughter of the Government commissary, who assisted at the experiments.

In this country we have for a long time refrained from licensing any explosive containing potassium chlorate, because such are so easily exploded by impact or friction. A favourite and highly practical test of the late Dr. Dupré, the Home Office adviser, was that of giving the explosive a glancing blow with a broom-stick on a wooden floor, chlorate explosives hardly ever stood this test. There was also but little inducement to make these explosives on a commercial scale, because potassium chlorate was dear, and therefore powders made therefrom would hardly have been able to compete with other equally or more powerful explosives. With the advent of electrolytic methods for the manufacture of chlorine, potassium chlorate and the like, chlorate explosives were brought within easy reach of the trade, and in

fact the present price of electrolytic potassium chlorate will under certain conditions permit the economical manufacture of suitable explosives Hence greater efforts were made to render chlorate explosives more stable, so as to pass the Home Office test, and ultimately success was attained by the addition of some oil Its function is evidently to so surround the potassium chlorate that, when mixed with carbonaceous matter, it becomes less sensitive The addition of greasy matter to chlorate explosives is not at all a new idea In 1867 already F. Hahn added spermaceti to a gunpowder containing chlorate.[1] In 1880 Tschirner [2] employed tar, and in 1882 Professor Himly [3] patented the use of a solution of hydrocarbons, which idea was again applied for by Lyte and Lewall in 1884.[4] Fraenkel in 1888 used naphthalene and paraffin,[5] Brank in 1891 took linseed oil,[6] and many other explosives containing hydrocarbons and tarry substances may be mentioned, which were the forerunners of the present-day chlorate explosives. However, a practical explosive was ultimately obtained in Cheddite, patented by Mr. Street,[7] and so called because it was first made in Chedd, in Switzerland. The more usual variety is known abroad under the name of Cheddite 60 bis, and its composition is 80 parts of potassium chlorate, 13 parts of mononitronaphthalene, 2 parts of dinitrotoluene and 5 parts of castor oil, whilst in this country the proportions of mononitronaphthalene and dinitrotoluene are reversed.

It is interesting to observe how the same old mixtures are proposed over and over again with slight alterations only, in order to qualify for a patent. Potassium chlorate with some carbonaceous matter like charcoal, sugar, starch, glycerine, flour, or sometimes a vegetable or mineral oil and the like occurs again and again. One patent [8] is of special historical interest, since it proposes the use of "Maltha" as an ingredient The patentees came from California, an English-speaking country, and therefore it might be supposed that the name was not unfamiliar in England, but this appears not to be the case I recollected, however, a passage in Roger Bacon's "Opus Majus" as follows "Nam Malta, quæ est genus bituminis et est in magna copia in hoc mundo, proiecta super hominem armatum comburit eum"—"Thus Malta, which is a kind of bitumen, and exists in large quantities in this world,

[1] British patent, No 960 of 1867
[2] *Id*, No. 447 of 1880
[3] *Id.*, No 1969 of 1882
[4] *Id*, No 14379 of 1884
[5] *Id*, No. 13789 of 1888.
[6] *Id*, No. 5027 of 1891.
[7] *Id.*, No. 9970 of 1897
[8] Quinby, Sharps and Greger, British patent, No 4781 of 1902

when thrown on an armour-clad man, burns him to death." It seems, therefore, that the *Mayflower* took with her some old-world expressions and adapted them to the new world.

The latest surprise is that in 1908 a chlorate explosive has been licensed as a safety explosive in this country under the name of "Colliery Steelite"; it consists of 74 parts of potassium chlorate, 25 parts of oxidized resin and 1 part of castor oil.

The electrolytic chlorine industry has also made possible the manufacture of pure perchlorates, and more especially of ammonium perchlorate, which presents many advantages, although the objection has been raised that explosives containing this ingredient generate fumes of hydrochloric acid in the mine. So far only Yonckite, a Belgian explosive, containing in addition to perchlorate, potassium nitrate and mononitronaphthalene, and a safety explosive made by the Carbonite Syndicate at Schlebusch under the name of "Permonite" have come into use.

Another class of explosives, which was from time to time used for ordinary blasting purposes, and of which very little has been heard in this country, are the Sprengel explosives. You have all heard of "Rackarock," which was employed in the blasting of the Hell Gate rocks near New York. Until the last decade it was hardly used anywhere except in America, but on building the first Chinese railways the Americans were able to introduce it.[1] It seems so simple to take powdered potassium chlorate, fill it into cartridges or linen bags, and then pour nitrobenzene on it. Nevertheless this would really be manufacturing an explosive either in the pit or near it, under conditions which could not be easily regulated, and therefore they could not be authorized in most countries In China and Siberia no such objection was raised, hence it is not surprising that a number of explosives were patented by Russian inventors, all of which closely resemble Sprengel's original explosives. One of these is now licensed in Italy.

A novel ingredient was introduced by Winand,[2] who mixes tetranitromethane with petroleum or other carbonaceous matter. Tetranitromethane (nitrocarbon CN_4O_8) crystallizes below 13° C. in white needles, and boils at 126° C.; it is not acid, does not mix with water, and might therefore be useful as an ingredient in other explosives.

Some of the Sprengel explosives are very powerful, and not

[1] Károly Gubányi, The Rackarock Blasting Powder, "Magyar mérnok és épitész egylet kozlonye," 1901, p. 165.

[2] British patent, No. 26261 of 1907.

without advantage, but it is always somewhat difficult to manipulate them with safety and cleanliness

A new departure was made in 1899, when Dr. Richard Escales of Munich invented the first aluminium explosive The use of manganese dioxide as an ingredient of black-powder-like mixtures for the purpose of augmenting the available oxygen has often been proposed, and, as can readily be understood, without success. In 1888 already Chapman patented the use of magnesium in cap composition,[1] in 1898 Weiffenbach of Munich proposed the admixture of aluminium to fireworks,[2] and in the beginning of 1899 Friese-Green and Knell vaporized magnesium in a gun by passing an electric current through the charge.[3] These were the only early attempts to utilize light metals in explosives, until Escales showed that the addition of aluminium or magnesium very considerably increased the temperature of explosion and thereby the explosive force. His explosive was patented under the name of Wenghœffer,[4] and is now I believe manufactured together with a similar explosive invented independently in 1900 by Ritter von Dahmen,[5] and since known under the name of "Ammonal"

Ever since aluminium has been taken as an ingredient in almost any kind of explosive. Theoretically it would be of very great value, but in practice the high price of aluminium powder and the possibility of oxidation under suitable conditions have somewhat militated against it. It is, however, used in Austria-Hungary for filling shells, for which purpose it seems well suited not having given any trouble during ten years of storage, although I am told they sometimes fail to explode. It is also on the special list of the British Home Office as an explosive for fiery mines.

Other metals might have a similar or even a better effect than aluminium. Thus in 1900 already Désiré Korda of Paris and the author have considered the possibility of using ferro-silicon, which can now be made up to 100 per cent. purity, and would readily oxidize. Its very great hardness, the difficulty of making it into a fine powder, and the possible danger arising from the presence of a hard, gritty substance, however finely divided, did not encourage further experiments. It has, nevertheless, been the subject of patents by others in 1904, 1905 and 1907, but so far I believe without success. In addition to the above-mentioned metals the use of iron, silicon, silicon carbide, zinc and its alloys, copper, and also the rare metals has been patented

[1] British patent, No 16997 of 1888
[2] *Id.*, No. 7579 of 1898.
[3] *Id.*, No. 11345 of 1899.
[4] *Id*, No 24377 of 1899.
[5] *Id*, No. 16277 of 1900.

In his patent of 1871 on the explosives bearing his name [1] Professor Hermann Sprengel, F.R.S., said, seemingly without reference to the rest of the patent, "I also employ picric acid," but in his famous lecture delivered in 1873 before the Chemical Society he said distinctly "Be it noticed here that picric acid alone contains a sufficient amount of available oxygen to render it, without the help of foreign oxidizers, a powerful explosive, when fired by a detonator. Its explosion is almost unaccompanied by smoke." As a matter of fact Sprengel did fire some shots with picric acid at Messrs John Hall & Sons' factory in Faversham in 1871, but was not encouraged by the Service to pursue his experiments.

Nothing further was heard of picric acid until 1886, when, as mentioned before, Eugène Turpin of Paris showed how to compress or melt it for use in shells The French service used picric acid, mixed with collodion to give it greater density, under the name of Melinite Later on it was compressed, but ordinary detonators failed to explode it with safety, and the expedient devised by Alberts and the author to use a primer of dry gun-cotton was too inconvenient The picric acid has therefore to be melted, in which state it can be more readily exploded by detonators, and has a density of about 1 65. Picric acid melts at 122·5° C., and must therefore be either heated in an oil-bath by high-pressure steam, or in a special "stove." Melting it at such a high temperature is very inconvenient and is not without danger, hence use was made of the well-known phenomenon, that a mixture of two substances of high melting-points has nearly always a lower melting-point than that of either of its constituents. Girard [2] has given a long list of the melting-points of explosive mixtures of this kind, which, coming from such an eminent chemist, deserves to be kept on record. A few characteristic mixtures are the following—

Mixture in equimolecular proportions of	M.P	M.P. of mixture.
Trinitrophenol Nitronaphthalene	122° C 61°	49° C.
Trinitrophenol Dinitrotoluene	122° 71°	47°
Trinitrophenol Trinitrocresol	122° 107°	70°
Trinitrophenol Trinitrocresol	122° 107°	78°
Trinitrocresol Trinitrophenol	107° 122°	80°

[1] British patent, No. 2642 of 1871. [2] *Id.*, No. 6045 of 1905.

Almost every country has adopted picric acid as a disruptive agent, under a different name, and differences in composition consist merely in the addition of an ingredient to reduce the melting-point. Such additions are nitronaphthalene, camphor, dinitrotoluene, etc., and the names are Melinite, Lyddite, Pertite, Shimose powder, Picrinit, Ecrasit, etc.

Besides having a high melting-point, picric acid is inconvenient in other ways. Left in contact with metals or oxides it forms very dangerous picrates, hence the necessity of varnishing the interior of shells, giving special protection to the primers, and generally taking the utmost precaution to prevent access of foreign bodies while the acid is in the molten state. Picric acid has an intensely bitter taste (which is still more pronounced in the inky black smoke of burning picric acid), and therefore its manipulation is not very pleasant. It also imparts a fairly fast yellow coloration to the skin, which in some parts has procured the nickname of "canary birds" to the workers in picric acid. (I have found that in one factory common salt was used for removing the yellow coloration from the skin, but why it should do so is not quite clear) When used together with other materials, it must be remembered that, being an acid, it is liable to displace other acids; for instance, it sets free nitric acid from nitrates, and therefore while picric acid might be useful for increasing the power of certain explosives, it would actually decompose them.

In order to obviate these drawbacks Hauff had proposed the use of trinitroresorcine [1] and the Chemische Fabrik Griesheim that of trinitrobenzene [2] and trinitrobenzoic acid.[3] These substances were not favourably received, but trinitrotoluene has within the last few years come very much to the fore, and also possesses a great many good qualities. Its melting-point varies between 72° and 82° C. It may be handled with almost perfect safety, if pure, does not give off noxious fumes on melting, is quite stable, does not combine with metals, and generally has no acid properties. Like picric acid it is only slightly soluble in cold water. It is slightly less powerful than picric acid, which is rather an advantage, since the latter frequently pulverizes a shell, instead of bursting it into a number of fragments sufficiently large to have destructive effect. Trinitrotoluene is very easily detonated. I have been able to explode it in the form of powder, with a No. 3 detonator only (0·540 gram of fulminate composition).

[1] British patent, No 9798 of 1894.
[2] German patent, No. 79477 of 1893.
[3] *Id*, No. 79314 of 1893.

Trinitrotoluene has been introduced into the French Servi under the name of Tolite. The Spanish Government call it Tril The Carbonite Works of Schlebusch are introducing it into oth Services under the name of Trotyl, and Messrs A. & W. Allendo of Schœnebeck under the name of Trinol, whilst other factor retain the name of Trinitrotoluol

According to Beilstein there are three isomers of trinitrotoluei the modification 1-2-4-6-trinitrotoluene [$C_6H_2CH_3(NO_2)_3$] resulti in large quantities, and being the one used Its melting-point, wh very pure, is 81° to 82° C., and this is demanded for military pı poses, but for use as an ingredient in blasting explosives, for whi it has found great favour, a lower melting-point (77° to 79° C.) sufficient The solidifying point of very pure trinitrotoluene 78¾° C. The 1-3-4-6-(β) and 1-2-3-6-(γ) modifications wi melting-points of 112° and 104° C respectively are not used, a special means have to be employed to eliminate them. On t other hand it is affirmed that in this country an 1-2-3-5 isom (not mentioned in Beilstein) is intentionally left in the finish trinitrotoluene by one factory, and the advantage claimed th thereby the fused mass solidifies more gradually, giving a high density on account of the reduced liability to form pores Th advantage can be secured also in other ways, and may be counte acted by a reduction of the melting- and solidifying-point resulti therefrom.

The manufacture of trinitrotoluene is carried out in stages, lil that of most aromatic nitro-compounds. Great care has to taken to purify the toluene, since that usually found in commeı contains benzene and other compounds. Nitration is effected enamelled iron vessels, and purification of the higher nitrat which cake together during nitration, has to be performed wi some care. Washing is usually completed in centrifugals. In ord to obtain the best quality, melting between 81° and 82° C., trinitr toluene made from purified toluene, and having a melting-point 77° to 79° C., is recrystallized from alcohol in vacuo. The machine for effecting this is not very complicated, but always special designed. In this country alcohol is somewhat dear and inco venient to use, in spite of facilities afforded for obtaining it du free, and petroleum benzene is therefore employed for recrystallizi the trinitrotoluene ; it is said, however, that a slightly darker coloı is imparted by this method to the product, to which objection taken in some countries.

From investigations made by Dr. Dupré and others, it is know

that the manufacture of nitro-compounds from benzene is to a certain extent inconvenient and even detrimental to health unless the precautions laid down in the Home Office regulations are taken. The manufacture of toluene nitro-compounds has to be effected under similar precautions, toluene and benzene being closely related as regards their properties. As is the case with all nitro-compounds, decomposition and even fires may occur during nitration if the reaction is allowed to proceed too vigorously. The dust from trinitrotoluene is not bitter, like that of picric acid

The density of trinitrotoluene, when loose, being 1·50 and when molten 1·600, means have been devised to increase it. Rudeloff[1] obtains a density of 1 85 to 1 90 by making a plastic substance from trinitrotoluene and potassium chlorate with a gelatine made from dinitrotoluene and soluble nitrocellulose. Bichel makes a plastic compound with collodion cotton, liquid dinitrotoluene and larch turpentine, calling it Plastrotyl.[2] Messrs Allendorff mix the trinitrotoluene, together with some lead nitrate or chlorate, with a gelatine made from dinitrotoluene and nitrocellulose, and call it Triplastit. This is an improvement on the way the French Government made Melinite with collodion, or Wolff & Co. filled cut gun-cotton slabs into shells with paraffin wax. Bichel also melts the trinitrotoluene, and after first exhausting all occluded air, compresses it by introducing compressed air above it[3] Bichel has in this way obtained densities up to 1 69. Rudeloff presses it in hydraulic presses under a pressure of 2000 to 3000 atmospheres, whereby it obtains a density of 1·7, and can be cut and worked like gun-cotton. For the purpose of facilitating detonation, some loose trinitrotoluene is used as a primer. Trinitrotoluene is also used in detonators, of which further mention will be made later on.

Another new explosive for filling shells is used in Spain under the name of Tetralit[4] It is said to be made from tetranitro-methylamine, and to be more sensitive than trinitrotoluene, but very little else is known.

The use of picric acid for filling shells has necessitated the use of a special primer, because picric acid cannot be detonated with certainty by ordinary detonators In this country picric powder is used for such primers, and is made of ammonium picrate and

[1] "Zeitschrift fur das gesamte Schiess- und Sprengstoffwesen," 1907, p 7.
[2] British patent, No. 16882 of 1906 [3] *Id*, No 19215 of 1906
[4] "Zeitschrift fur das gesamte Schiess- und Sprengstoffwesen," 1908, p. 308.

potassium nitrate. This is nothing but Brugère's powder of the nineteenth century.[1]

During the last three or four years newspapers contained accounts of trials with a new explosive, at first called Vigorite and now Bavarite, the invention of Professor Schulz and Mr. Gehre, which is said to cost only one-third as much as other explosives, and to be ever so much more powerful. On examining the patent[2] one finds that this is nitrated solvent naphtha, a somewhat indefinite mixture of various compounds, giving on nitration a mixture of nitromesitylene, nitrocumol, and other substances. The inventors have, in a later patent,[3] indicated means to purify the solvent naphtha, and the new nitro-compound made therefrom is evidently now on trial in Germany. It must be embarrassing to the inventors to see such improbable accounts of manufacturing costs and exaggerated effects produced by the explosive, published in newspapers.

[1] *Comptes rendus*, vol 69, p 716.

[2] British patent, No. 5687 of 1905.

[3] *Id*., No. 19565 of 1907.

II

I HAVE mentioned in my previous lecture that Sobrero invented nitroglycerine in 1847. It is known that, although he recognized the value of this invention for civil and military blasting purposes, practically no use was made of it until 1867, when Alfred Nobel invented dynamite, and was not deterred by accidents and prejudice from introducing it into the service of mankind. You know that before this time Mowbray in Massachusetts manufactured nitroglycerine and carried it into the mines in the frozen state.

Nobel devised processes for the manufacture of nitroglycerine on a large scale, and the machinery for it was constructed to his ideas by his life-long adjutant, Mr. Alarik Liedbeck of Stockholm. Since there is a full description of all the apparatus in use in my book on "The Manufacture of Explosives," which appeared in 1895, I can confine myself to dealing with progress made since that date. You will find described in this book two kinds of apparatus for nitrating glycerine, such that have a helical revolving stirrer for mixing purposes, and such that are agitated by compressed air. Occasionally both mechanical and compressed air stirring are used. One has learnt in time to control the operation of nitration more efficiently, and this inspired confidence to increase the size of the apparatus. I believe the largest apparatus made in lead, nitrates 680 kgs. of glycerine at one operation, whilst in America and South Africa steel apparatus with mechanical stirring gear are mostly used, some nitrating 1000 kgs. at a time. In one United States works they have gone so far as to have four such steel nitrators, each for a charge of 1000 lbs. of glycerine, in one room, and driven from one main shaft, but present practice is to have two such nitrators in one building In this country one would not allow more than one nitrating apparatus to be used at a time. Of course each nitrator is provided with a series of lead or steel coils through which cold water circulates, and it has now become frequent to install a refrigerating plant and to circulate water of only 10° C. and less through the coils

Twenty years ago the recovery of glycerine from soap lyes had

just begun, most of the glycerine used being so-called "refined," which was a fairly pure material, coloured dark by cell substance that had been slightly burnt in the stills. Now-a-days practically all glycerine used is "distillation," recovered by neutralizing the soap lyes and distilling off the glycerine contained therein. It is almost chemically pure, and a substance of reliable uniformity.

With regard to the composition of the nitrating mixture, it has been customary in well-conducted factories during the last twenty years or so to nitrate 110 kgs. of glycerine in a mixture of 300 kgs. of nitric acid of about 93 to 94 per cent. monohydrate and 500 kgs. of sulphuric acid of 96 per cent. monohydrate (and not, as Sir Fred. Nathan and Mr. Rintoul stated, 100 parts of glycerine and nitric acid of 91 per cent. only)[1] This corresponds to about 255 parts of nitric acid monohydrate and 436·4 parts of sulphuric acid monohydrate, or a total of 691 4 parts of acid monohydrate with 35·8 parts of H_2O (4 9 per cent.) to each 100 parts of glycerine.

Some sixteen years ago Messrs. Chapman, Messel & Co. and the Badische Anilin- & Soda-Fabrik tried to introduce sulphuric acid anhydride for the manufacture of explosives. At that time the price of anhydride was still such as to preclude its use for strengthening up the waste sulphuric acid of about 1 600 obtained in denitrating waste acids, and it was also considered impracticable to add it to the waste acid itself. About eight years ago, however, in France and elsewhere, mixtures of nitric and sulphuric acids of high strength, obtained by the addition of sulphuric anhydride, were used for nitrating glycerine, and as soon as the processes for manufacturing sulphuric anhydride were no longer kept secret, but became known through the patents which were at last applied for, a number of explosive factories erected such works. It is now customary to add sulphuric acid containing 20 per cent. of anhydride (oleum) to the original mixture, but it is still found impracticable to add it to the waste acid. It will be seen from the paper of Sir Frederic Nathan and Mr. Rintoul on "Nitroglycerine and its manufacture" that the use of anhydride has reduced the quantity of sulphuric acid required. Five years ago already I found in the Pozsony factory of Nobels' the use of mixed acid consisting of 37·2 per cent. HNO_3, 60 per cent. H_2SO_4, and 2 8 per cent. H_2O, and made with anhydride. Although no artificial refrigeration was used the yield of nitroglycerine amounted to 220 for 100 glycerine and a ratio of 6·318

[1] "Journal of the Society of Chemical Industry," March 16, 1908. Compare also Guttmann, "Manufacture of Explosives," vol. ii, p. 93.

of acid to 1 of glycerine Factories using Nathan, Thomson and Rintoul's process now employ a mixture of 41 per cent. HNO_3, 57·5 per cent. H_2SO_4 and 1·5 per cent. H_2O, corresponding to 250 lbs. HNO_3, 350 lbs. H_2SO_4 and 9 lbs. H_2O for each 100 lbs. of glycerine, which gives a ratio of 6 09 of acid to 1 of glycerine, as against 6 91 to 1 formerly required. It is thus seen that this process requires about the same quantity of nitric acid per 100 glycerine as the old process, but about 86 lbs, or roughly 20 per cent., less sulphuric acid. It will therefore simply depend upon the price of the sulphuric anhydride whether it is advantageous to use it.

With the present prices of £3 per ton of 96 per cent. sulphuric acid and £3 15*s.* 0*d.* per ton of sulphuric monohydrate, containing 20 per cent. of anhydride, the difference between the cost of materials with the former yield of 220 and the present one of 229 nitroglycerine, is shown in the following calculation—

OLD PROCESS.	£	s.	d.
1·10 tons of glycerine at £50	55	0	0
3 00 ,, 93 5 per cent nitric acid at £20	60	0	0
5·00 ,, 96 per cent. sulphuric acid at £3	15	0	0
Yield 2·42 tons of nitroglycerine	130	0	0
Cost per ton	£53	14	5

NEW PROCESS.	£	s	d.
1 00 ton of glycerine at £50	50	0	0
2¾ tons of 91 per cent nitric acid at £19 10*s.*	53	12	6
3½ ,, H_2SO_4 with 20 per cent. SO_3 at £3 15*s*	12	10	0
Yield 2·29 tons of nitroglycerine	116	2	6
Cost per ton	£50	14	3

Difference per ton, £3 0*s* 2*d* or approximately 5 6 per cent.

This apparent saving is quite counterbalanced by the fact, that in the new process 1·9 tons less of waste acid are obtained.

It would, therefore, seem that the price of anhydride ought to be lowered, considering that the makers actually dilute the anhydride in order to make acid of 100 per cent. strength. In making this comparison it must, however, be remembered, that with the new process the same apparatus will hold 18 per cent. larger charges

After nitration the mixture is allowed to stand, when the nitroglycerine separates from the waste acid and floats on the top of it. This operation is usually performed by running the nitrating

mixture into a separate vessel having a conical or inclined bottom, the so-called separator, from which the nitroglycerine is taken off either by means of a skimmer or by earthenware cocks placed at about the level where nitroglycerine and waste acid meet. The separation is sometimes considerably delayed by the formation of a silicious colloid, which agglomerates with particles of cell substance and other impurities, forming fern-like growths The Dynamit Actiengesellschaft in Hamburg[1] found a very efficient means of promoting separation in the addition of high boiling paraffins in quantities of 0 5 to 2 per cent. of the weight of glycerine, whilst Dr L F Reese of Wilmington,[2] adds as little sodium fluoride as 0 002 per cent. (1 in 50,000) of the glycerine employed to the nitrating mixture with excellent results. Both methods are now used in very large factories. For more than thirty years some factories had been in the habit of employing one vessel only for both nitrating and separating, and withdrew the nitroglycerine from three earthenware cocks, placed at short intervals at the separating line. This enabled them to gain considerably in levels and to carry out the manufacture right up to the final washing on practically the same level.

The waste acid was always sent to after-separation houses, which were frequently called by the German name of "Nachscheidung." They were designed by Mr. C. Gœpner in 1882. The acids were kept therein in large lead vats, having a dome-shaped cover carrying a glass tube fitted with a cock or stopper at the side. Cooling worms kept the temperature down, and the nitroglycerine which separated out was displaced by adding some waste acid from a tank on a higher level until the nitroglycerine rose in the glass tube and could be withdrawn through the cock or tubulure. Since the waste acid sometimes had to be kept in these after-separation houses for a week, in order to get rid of all the drops of nitroglycerine which separated out, decomposition occasionally set in. A very small number of factories therefore adopted the plan of placing the waste acid in a lead tank, standing apart from other buildings, and tipping into it water from a small tank, pivoted in a suitable manner. The heat generated by the sudden addition of water decomposed the waste acid, and the nitric acid was driven off. Whilst this was certainly an efficient, if somewhat risky, method of decomposing all the nitroglycerine contained in the waste acid, it was also wasteful in causing the loss of all the nitric acid. A better plan was thereupon introduced in France and elsewhere,

[1] British Patent, No. 13562 of 1904. [2] *Id*, No. 20310 of 1905.

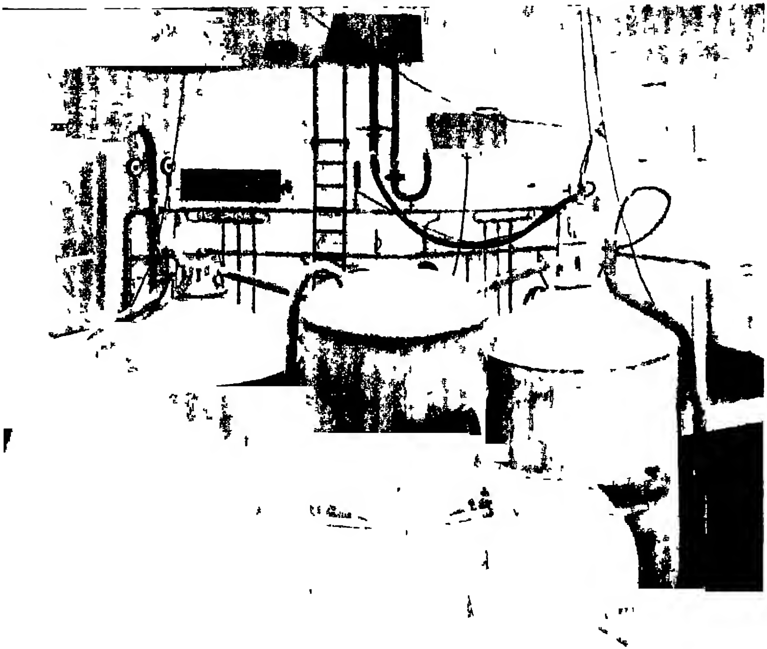

FIG. 1.—NITRATOR SEPARATOR (NATHAN, THOMSON AND RINTOUL'S PATENT)

[To face p. 21

which consisted in gradually diluting the waste acid by the addition of from 2 to 3 per cent. of water, thereby stopping the further formation and separation of nitroglycerine.

At the Government Factory at Waltham Abbey these methods have been improved upon. A so-called nitrator-separator is used, in which the nitroglycerine has time to separate from the acids, and waste acid is then added from below, thereby bringing the level of the nitroglycerine to a point where it will run out through a gutter into the preliminary washing tank. In this way the use of cocks is avoided. When all the nitroglycerine has been displaced about 2 per cent. of water are introduced gradually, to avoid undue heating of the mixture, further formation and separation of nitroglycerine thus being prevented. Sir Frederic Nathan and Mr. W. Rintoul describe in their above-mentioned paper how they investigated the conditions under which nitroglycerine was absorbed by the waste acids.

The result of this combination of a number of useful processes, namely, the employment of anhydrous sulphuric acid to produce a mixed acid containing little water, the use of refrigerated water to cool the acids, the displacement of the nitroglycerine by means of waste acid, which obviated the remixing of acid and nitroglycerine on emptying the nitrator, and the addition of water to stop the separation of further quantities of nitroglycerine, was that they together contributed to yield much better results. As a matter of fact in well-conducted works the yield of nitroglycerine with the proportions of 6·91 to 1 mentioned above was between 217 and 220, at Waltham Abbey it was possible to obtain by the "displacement process" a yield of 229 parts nitroglycerine for 100 parts glycerine, instead of the former 220 parts. According to Mr. de Mosenthal the Nobel works obtained similar good results. This yield has to the author's knowledge been only once exceeded in a Belgian factory, when a charge of nitroglycerine had to be drowned on a cold winter's day. The contents of the tank froze and required two days to thaw; a yield of 240 parts nitroglycerine was, however, the surprising result. It is a fact, also, that by this process the nitration, first separation and first washing can all be done on the same level, whilst no after-separation house and apparatus are required. The drawback of the system is, that the number of apparatus required is no smaller than before, because whilst one nitrator-separator is at work, no other apparatus may be used in the same building, and the separation takes a good deal more time than before on account of the several manipulations required. With the nitrator-separator

apparatus formerly in use, where the nitroglycerine is drawn off through cocks, about 30 inches more head are required for the whole system. Certain other works were able to further reduce the difference of level by drawing up the prewashed nitroglycerine into a final washing tank by means of a previously evacuated vessel. The author has constructed one of these in such a way that no cock whatever was necessary except for the vacuum pump.

With regard to the selection of apparatus, round lead or steel tanks as explained above, are generally used, but the author has also seen square-cornered ones, which seem to present some advantage, their oblong form making it easier to arrange the coils in them, while both the entrance of glycerine and that of compressed air can be arranged for in several places; better mixture is secured thereby. The Americans are much in favour of mechanical stirring, whilst in Europe air stirring is preferred. Having worked with both I cannot see much difference as regards results, but since I do not like to have any moving parts in connection with the manufacture of nitroglycerine, I think air stirring is preferable on the whole.

There has been no special improvement in the manufacture of dynamite since Nobel in 1875 invented blasting gelatine. This and the gelatine dynamites made by mixing a thin blasting gelatine with an absorbing powder of potassium nitrate and wood meal, have in most countries driven kieselguhr dynamite out of the field. As a matter of fact scarcely a few tons of such dynamite are sold in either Germany or Austria, but a certain quantity is still produced in this country.

In this connection it will be interesting to have a true picture of kieselguhr as used for dynamite. Mr. Henry de Mosenthal, whose skill in preparing specimens for the microscope we had often occasion to admire, has prepared for me various slides of kieselguhr, which has undergone the usual calcining process in manufacture, and which I selected on account of its high absorbing capacity (80 to 82 per cent of nitroglycerine). A good deal of care was required in photographing this specimen, as a number of characteristic diatomes had to be cut out and combined into a new photograph, but I think the result was well worth the trouble.

For blasting gelatine, as you know, a so-called collodion cotton or soluble nitrocellulose is employed. On account of the very stringent regulations in this country, and more especially in Australia and South Africa, it is necessary to use a special collodion cotton which will not only be completely soluble in nitro-glycerine,

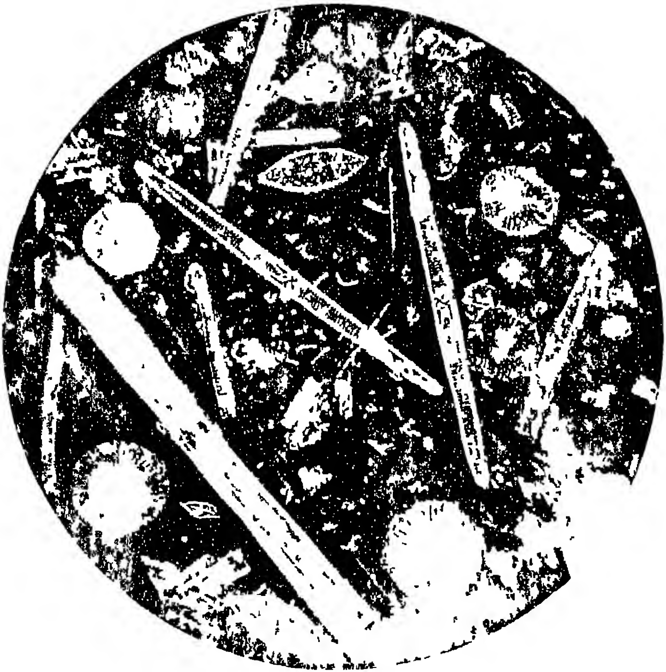

FIG. 2.—CALCINED KIESELGUHR

[To face p. '

but will efficiently retain it under any climatic conditions. The selection of such collodion cotton is not an easy matter, and it will generally be found that an inferior nitrocellulose will not produce a good gelatine, however much of it is put in. Many people think that if 7 per cent. of nitrocellulose are insufficient to make a stiff and suitable blasting gelatine, the addition of another 1 or 2 per cent. would do it, and certainly at first the resulting gelatine is so stiff and hard as to require special effort in the cartridge machines. After a few months of storage, however, or after passing over the equator into Australia, nitroglycerine is found to exude. A good nitrocellulose will give a perfectly stiff blasting gelatine, with between 6 and 7 per cent. of nitrocotton, and if a 2½ per cent. solution is made in a porcelain basin, the resulting gelatine should be easily detachable after cooling, showing no signs of exudation.

If no improvements have been made with regard to increasing the strength or altering the composition of dynamite to obtain better results, there has been within recent years a revival of old ideas, but with better success, for the purpose of obviating one of the chief objections to dynamite, namely, that of freezing. It was in 1866, in Sweden, that A. E. Rudberg patented the addition of nitrobenzene to nitroglycerine for the purpose of making it unfreezable.[1] This patent being in a language hardly understood elsewhere, and at that time no printed specification having been published in Sweden, the patent was so forgotten that Nobel, a Swede himself, patented it again in 1885.[2] I had myself worked on the same subject in conjunction with Nobel, but I found that most additions to nitroglycerine reduced the explosive power considerably, when used in such quantities as to be efficient. The Société des Poudres et Dynamites, of Arendonck, found later,[3] that the addition of dinitrotoluene dissolved in nitroglycerine was very useful in lowering the freezing point. A new departure was really made when Dr. Anton Mikolajczak in 1904 patented the addition of dinitroglycerine to trinitroglycerine explosives, and at the same time indicated a practical method of manufacturing it.[4] It is now made on a large scale in a factory at Castrop, in Germany. Liecke mentioned already in 1865 that he obtained mono- and dinitroglycerine by nitrating pure glycerine at 0° in a mixture of 1 volume of nitric acid of 1·4 specific gravity and 2 volumes of concentrated sulphuric acid.[5] In the German patent, No. 58957,

[1] Swedish patent, April 30, 1866. [2] British patent, No. 5330 of 1886.
[3] *Id.*, No. 14827 of 1903. [4] *Id.*, No. 8041 of 1904
[5] "Mittheilungen des Hannoveranischen Gewerbe Vereines," 1865, p. 214.

of 1890, Wohl described some properties of mono- and dinitroglycerine, and mentioned their suitability for reducing the freezing-point of nitroglycerine. In order to understand the question better it is necessary to point to a most interesting work by Sigurd Nauckhoff,[1] showing why nitroglycerine can sometimes be subjected to intense cooling without freezing (supercooling), and to a paper by Dr. H. Kast[2] showing that there are two kinds of nitroglycerine (one being an allotropic modification), with two different melting-points, one nitroglycerine solidifying at about 13·2°, and the other at about 2·1°, the melting-points being 13·5° and 2·5° respectively.

Professor Will, in an investigation on glycerine nitrates,[3] has given an historical *résumé* of the invention of dinitroglycerine, and made extensive experiments on the use of mono- and dinitroglycerine. Without going closely into the matter, which is best read up in Will's original paper, the conclusion to which he arrives may be mentioned, namely, that dinitroglycerine is not a sure guarantee against solidification, and that under certain conditions explosives prepared with it may become solid at a higher temperature than trinitroglycerine explosives.

Since dinitroglycerine is somewhat hygroscopic, and its manufacture rather more expensive than that of trinitroglycerine, a number of other inventors have tried to find suitable substitutes for the prevention of solidification. In the first instance, the substitution of a hydroxyl group in the glycerine by hydrochloric acid was tried, monochlorhydrine being obtained. From this the Westfalisch-Anhaltische Sprengstoff-Gesellschaft made dinitromonochlorhydrine and mononitrodichlorhydrine.[4] On the other hand glycerine is polymerized by heating it with concentrated sulphuric acid, whereby diglycerine is obtained. This can also be obtained by the prolonged heating of glycerine without acid to between 290° and 295°. If some diglycerine is added to ordinary glycerine, and the two nitrated together, a mixture of trinitroglycerine and dinitro- or tetranitrodiglycerine is obtained, which prevents freezing.[5] The Deutsche Sprengstoff-Gesellschaft has patented monochlordinitroglycerine,[6] whilst Escales (Lehmann) prepares a complex product of various chlorhydrines and diglycerines and nitrates the mixture.[7] Another addition was

[1] "Zeitschrift fur angewandte Chemie," 1905, p. 11
[2] "Zeitschrift f d ges. Schiess u. Sprengstoffwesen," 1906, p 225.
[3] "Berichte der deutschen chemischen Gesellschaft," 1908, p. 1107.
[4] British patent, No 4057 of 1906. [5] *Id.*, No. 6314 of 1906.
[6] *Id.*, No 14958 of 1905. [7] *Id.*, No. 21117 of 1907.

proposed by Vender, who prepared dinitroacetine and dinitroformine.[1]

Of all these additions none has so far been definitely adopted for the manufacture of unfreezable dynamites, but, I believe, that lately dinitrodichlorhydrine has been used with considerable success by the German works of the Nobel Companies.

With regard to safety explosives containing nitroglycerine I shall have more to say when speaking about safety explosives in general

We now come to gun-cotton. The history of the invention of gun-cotton has been told so often, and the steps that led up to its successful manufacture during the last forty years have been so often detailed, that I need not refer to them at any length The really important step in the manufacture of gun-cotton was taken when the British Government adopted a process of pulping and purifying the gun-cotton, first patented by John Tonkin, jun., of Poole, near Copperhouse, in Cornwall,[2] and again, in combination with the compression of the pulped gun-cotton, three years later by Sir Frederick Abel.[3] The next step was made when the principle of the detonation of nitro-compounds by means of a small fulminate of mercury charge, invented by Alfred Nobel,[4] was extended by Mr. Brown, Sir Frederick Abel's assistant, to gun-cotton.[5]

The British Government gave the German Government an opportunity of inspecting the gun-cotton works at Waltham Abbey, and supplied them with plans for the erection of a similar factory, which is still in existence in Kruppamuehle in Upper Silesia.

Baron von Lenck, the Austrian general, who worked most assiduously as the pioneer of Schonbein's invention, used gun-cotton in hanks; the British Government introduced the use of cotton-waste from spinning and other operations where threads are made. The reason for this change is not quite apparent, unless it was felt that since the cotton had to be pulped in any case the cheaper waste might do just as well as the long threads. This use of cotton-waste has continued ever since. Originally it was boiled in a 2 per cent. sodium carbonate solution only and then carefully washed; later on caustic soda was used, and with the advent of smokeless powders very stringent regulations were drawn up as to the amount of fat permissible in the cotton, and great importance

[1] British patent, No 9791 of 1906.
[2] *Id.*, No. 320 of 1862.
[3] *Id.*, No. 1102 of 1865.
[4] *Id.*, No. 1345 of 1867.
[5] *Id*, No 3115 of 1868

attached to the cotton being white and clean. As a result, large cotton bleaching works have been established, in which the cotton is boiled with caustic soda for the removal of fat, then bleached with bleaching powder, and after washing neutralized with sulphuric or hydrochloric acid, the calcium sulphate or chloride formed being carefully washed out again. Other works bleached with calcium sulphide or similar strong bleaching agents. The cotton so treated is dried, and either sold in this state or else passed through a willow or similar carding machine. In the latter case it has been freed from nails, wire, and other accidental admixtures which are generally found in cotton bales. For insoluble gun-cotton as used in torpedoes, shell charges, etc., uncarded cotton-waste is bought in this and several other countries, whilst for collodion cotton carded and very white cotton is generally preferred.

It is very curious that in the purchase and use of nitric and sulphuric acid for the nitration of gun-cotton, most stringent conditions are laid down with regard to freedom from mineral matter, chlorine, sulphates, arsenic, etc. Yet, as far as I could ascertain, no special precautions seem to be taken in the case of cotton to guard against impurities. The conditions of supply in this country simply demand a certain small maximum of fat, and reliance is placed upon practical tests. In other countries they go even so far as to ask for the cotton to be white and free from iron parts. As a matter of fact uncarded cotton-waste as used for gun-cotton generally contains a quantity of strings, wicks, coloured threads, india-rubber, or elastic cords, and similar leavings, showing the origin of the waste, and no amount of hand picking can free the cotton absolutely from such impurities. I have further found in cotton supplied by manufacturers of the best repute a large amount of chlorine, sulphate of lime and sulphides, besides organic and mineral dust, which gives the cotton quite a grey appearance.

Is it not also strange that it never occurred to anybody—at least as far as I know—to ascertain whether the impurities in the cotton, brought about by forcible treatment with bleaching agents and acids, are responsible for a great deal of the instability of certain finished gun-cotton and smokeless powders? I am convinced that this is the case. It is true that Cross, Bevan and Jenks,[1] as well as Lunge and Bebie,[2] have compared the nitration

[1] "Journal of the Society of Chemical Industry," 1900, p. 318.
[2] "Zeitschrift für angewandte Chemie," 1901, p. 483.

of raw cotton with that of bleached cotton, but only as far as yield, solubility, and combined sulphuric acid are concerned, nobody, however, seems to have given it a thought that such a complex compound as cellulose in the shape of cotton must vary to an enormous extent, both in its physical and its chemical structure, and thereby also the nitrocellulose made from it, thus requiring far more control than the mere percentage of nitrogen and the solubility and viscosity may involve.

Let us examine the possible changes. In the first instance we have the cotton itself, which may be in any stage of ripeness It is well known that the riper the cotton the better the fibre, and the easier it takes dyes, so much so that there is what is known as dead cotton, which causes spots in dyed fabrics. It has frequently been examined under the microscope, but quite recently Dr. R. Haller[1] has investigated this dead cotton by means of ammoniacal copper solution, caustic soda, etc. Such cotton is, according to F. H. Bowman,[2] always insufficient in strength, and breaks up in the manufacturing process. Haller found that it dissolves with difficulty in ammoniacal copper solution, is only coloured slightly yellow by iodine-potassium-iodide solution, and shows no brilliancy in polarized light. Only fully matured cellulose gives normal reactions.

The investigations of Leo Vignon[3] on the formation of oxycelluloses and hydrocelluloses, and the behavior of their nitro-compounds, show plainly how cotton and cotton-waste may, by the nature of the treatment they undergo, be partly transformed into oxycellulose, which gives an unstable nitro-compound, and into hydrocellulose, which has a different rate of nitration than ordinary cellulose.

I have repeatedly stated on previous occasions that in my opinion the process of nitration with a mixture of sulphuric and nitric acids results in the first instance in an attack on the cotton by the sulphuric acid similar to that in the manufacture of vegetable parchment, and that the sulphuric acid is gradually displaced by the nitric acid penetrating the fibre. It is obvious that according to the quantity and the strength of the sulphuric acid present in the mixture, nitration will proceed more or less rapidly and thoroughly, and that consequently the nature and the character of the nitrocellulose may be thoroughly altered

[1] "Chemiker Zeitung," 1908, p. 838.
[2] "The Structure of the Cotton Fibre," London, 1908, p 114.
[3] *Comptes rendus*, June 6, 1898, September 10 and 17, 1900.

according to the proportions and strength of acids used. Lunge and his collaborators have shown this influence as far as it can be shown in nitrating small quantities of medicinal cotton, which is not suitable for nitration on a large scale

It seems a fact that the more oxycellulose is formed in the cotton before nitration, the more unstable are the compounds formed in the nitrocellulose. Other impurities in the cotton are all the more likely to endanger the stability of nitrocellulose, as their nature is always unknown, and varies from sweepings to india-rubber elastics, while almost all are sure to produce unstable compounds.

How far the nature and origin of the acids may have an influence upon the ultimate product has still to be investigated. It is by no means impossible that the system of revivifying waste acids by means of sulphuric anhydride, which is now much in vogue, may from the nature of the process of the latter's manufacture introduce certain risks, more especially as it always contains some sulphurous acid. It is also known from the investigations of Will that the waste acid from the manufacture of gun-cotton generally contains nitro-compounds of various sugars, some of which are highly unstable, and it was found in Waltham Abbey that the use for the manufacture of nitroglycerine of nitric acid, made with waste acids from gun-cotton manufacture, was impairing the stability of nitroglycerine. It is, therefore, quite conceivable that the origin of the nitric acid may have an important bearing upon the stability of nitrocellulose, although it is said that nitric acid made with gun-cotton waste acid does not affect the nitrocellulose made therewith in an adverse manner

I do not think that differences in apparatus used for the manufacture of nitro-cellulose have much to do with its stability. There certainly is a difference in the amount of ash according to whether an iron, lead, or earthenware apparatus has been used, and it is quite conceivable that the solubility and viscosity may be influenced by the method of dipping and nitrating. To what extent the presence of iron salts formed in iron apparatus affect the stability is still a problem worth investigation. I have strong reasons for not recommending iron vessels for stabilization in the first instance. Considering all that I said above, I believe that if one must use nitrocellulose, and if, as seems to be the case, cotton is the best material for making it, one ought to use the natural cotton only, and not common yarn, and less still waste, which have both undergone so much forcible, mechanical and chemical

treatment as to completely alter the character of the cellulose, and introduce elements of uncertainty and danger. These should be avoided by the use of ripe raw cotton, which, of course, would have to undergo suitable treatment to eliminate fat, husks, and other impurities, but would not necessitate the whole bleaching operation with its attending defects.

The selection of the raw cotton is so far carried out purely on practical lines by nitrating various samples. Besides cotton, blotting-paper made from cotton and paper shreds were recommended, also tissue-paper, cellulose as used for paper-making, and other raw materials. All these cotton substitutes were, however, discarded, and even the celluloid and artificial silk industry use cotton Soda cellulose is taken for Schultze powder and some nitrocelluloses of minor importance

The cotton is generally hand-picked and opened in a willow, the better varieties of which are provided with a fan to draw off the fine dust formed It is then dried to about 0·5 per cent. of moisture. Some factories use a drying machine.

Formerly the mixture for gun-cotton consisted of 1 part of 1·500 nitric acid and 3 parts of 1·840 sulphuric acid, and each charge was revivified by taking away one-quarter of the waste acid and adding a mixture rich in nitric acid, so as to obtain about the original composition. The following table shows the result of revivifying the waste acid ten times in a series of operations made in 1886 by Dr. Abelli and the author—

No	Composition of nitrating mixture. Proportion 1 40			Temperature of nitration.	Yield	N	Soluble.	Composition of waste acid.		
	H_2SO_4	HNO_3	H_2O		%	%	%	H_2SO_4	HNO_3	H_2O
1.	72 82	24 37	2·81	20°	146 25	13 32	3 60	75·15	19 00	5 85
2	71 82	23 00	5 18	10	167·50	13 34	2 10	76 00	18 40	5 60
3	72 45	22 52	5 03	14	169·00	13 39	7·20	73 40	19 10	7 73
4.	70 21	23 05	6 74	10	165 75	13 49	2 93	71 40	20 22	8·38
5	68·77	25·97	7 26	12	175 00	13 38	2 88	71 06	20 51	8 43
6	69·47	23·40	7 32	10	166 25	13 08	2 26	71 72	19 48	8 85
7	70 00	22 34	7 66	10	165 00	13 40	4 00	71 71	18 82	9 47
8.	70 00	21 86	8 85	9	152 50	13 30	4 80	70 70	19 35	9 95
9.	69 18	22 58	8 24	6	167·50	13 22	1 60	71 00	19 13	9 87
10	69 40	22 00	8 60	9	152·50	13 21	3 46	70 00	19 00	11 00

The original mixture consisted of 1 part of nitric acid to 3 parts of sulphuric acid, both of over 97 per cent. monohydrate. Three parts of waste acid were revivified, with 1 part of fresh acids.

It will be seen that the percentage of nitrogen contained in the

nitrocellulose reaches a maximum when the percentage of water in the acid mixture is about 9 per cent., and not, as might be supposed, in the stronger acid. This observation led several factories to study the influence of water on the amount of nitrogen in and the solubility of the nitrocellulose, and it was found that thereby it was possible to secure uniform and predetermined results. Lunge and his pupils have made this and various other influencing factors the object of extensive investigations, and their results are a most valuable guide to manufacturers, although they cannot be translated directly into practice.

The majority of factories prepare the nitrating mixture by giving special consideration to the percentage of water in the first instance, because, by varying this, nitrocelluloses of widely different properties can be obtained I have often said that by varying the concentration of the acids, their temperature and the time of nitration, one has three factors, each of which can to a certain extent influence every property of the nitrocellulose obtained. Lunge and his pupils have shown by their researches what law obtains for every step in the alteration of each of these factors. To quote only a few instances By varying the percentage of water and the ratio between nitric and sulphuric acid, Sir Henry Roscoe showed in the cordite case that he obtained a soluble and an insoluble nitrocellulose, the one with 12 73 per cent. and the other with 12·83 per cent., or practically identical percentages of nitrogen. As shown in the previous table, an increase in the percentage of water up to a certain point rather tends to give the nitrocellulose a higher percentage of nitrogen, but without increasing the percentage of soluble nitrocellulose contained therein. If, however, the percentage of water exceeds 9 per cent., more and more soluble nitrocellulose is formed, until the transition into a wholly soluble nitrocellulose takes place fairly rapidly. It is the custom in a majority of factories to produce soluble nitrocellulose by taking equal parts of nitric acid of 75 per cent. monohydrate and sulphuric acid of 96 per cent. monohydrate and nitrating the cotton at a temperature of 40° C This nitrating acid therefore contains 14·5 per cent. of water, yet by merely altering the proportions of acid it is quite possible to make very good soluble nitrocellulose in the cold, and some modern factories make it in this way. It seems to be very difficult, if not impossible, to obtain good and stable completely insoluble nitrocellulose from wood pulp. On the frontispiece is shown a micro-photograph of ordinary and nitrated cotton fibres, as seen in polarized light. The slide was

prepared by Mr. Henry de Mosenthal, to whom my hearty thanks are due for this and other assistance with the microscope.

It is now recognized on all sides that there are no definite stages of nitration in nitrocellulose, but that the change in composition goes on without a break, if the conditions are suitable. Thus, for instance, the treatment to which gun-cotton is subjected in the course of stabilization has a great deal to do with its ultimate composition. Bruley[1] has already shown that prolonged boiling makes the gun-cotton more soluble, and also reduces the percentage of nitrogen. Excessive pulping also affects the solubility, whilst long treatment with even such feeble alkalies as calcium carbonate tends to hydrolize the nitrocellulose, and in any case decomposes oxycellulose. The manufacturer of gun-cotton and nitrocellulose is, as a matter of fact, face to face with great difficulties Almost everything he does tends to act detrimentally. From the nitration his nitrocellulose contains a number of lower nitro-compounds, nitrated oxy- and hydrocellulose, nitrosaccharoses, etc., which he has to get rid of. The usual way to do this is to boil the nitro-cotton for a long time, and when by the application of the well-known potassium iodide test the nitrocotton is shown to be reasonably free from such admixed impurities, it undergoes further treatment by means of pulping. This is the process as carried out at Waltham Abbey, whilst in other factories the cotton undergoes a further treatment after pulping. It is not quite clear why one should keep on boiling the long and closed-up fibres of unpulped gun-cotton for, say, 50 hours, as is done in some factories One would imagine that if after a preliminary washing or boiling the gun-cotton were pulped and then boiled, this could be done much quicker. As a matter of fact I have found that by heating the gun-cotton whilst pulping, the increase in stability is very much accelerated, and several factories use the method with advantage. In France they boil for 100 hours, and I have quite lately seen nitrocellulose that was boiled for 200 hours, without, however, being much the better for it. It must, however, be mentioned, that the Waltham Abbey gun-cotton, as at present made, is a very stable and good gun-cotton, as judged both by the iodide test and by the destructive test, of which more will be said later on. This is due, in the first instance, to an investigation carried out by Dr. Robertson. He showed that the former method of giving short boilings of two hours and following them up with long boilings of 8 and 12 hours was erroneous, and that two long

[1] 'Mémorial des Poudres et Salpêtres," 1895–6, p. 131.

boilings of 12 hours each would liberate acid from the nitrocellulose, giving an acid water which hydrolizes all the impurities without attacking the gun-cotton itself, and that subsequent short washings are useful in eliminating the products of hydrolysis. Having had frequent occasion to put Dr. Robertson's principles to a practical test, I consider it to be one of the most useful pieces of work accomplished since the invention of gun-cotton.

The nitration of the gun-cotton had originally been performed in iron dipping-pans, some fresh acid being added from time to time, and the dipped cotton, after squeezing out the bulk of the acid, left to complete the nitration in small earthenware pots. Later on nitration in large pots or cast-iron pans holding up to eight or more kilogrammes of cotton was carried out with carefully-prepared and analyzed mixtures of acid, which were revivified by properly analyzing the waste acid and adding the necessary revivifying acid. Messrs. Selwig and Lange of Braunschweig have, as is known, invented the so-called nitrating centrifugal machine, wherein the cotton is dipped and allowed to stand for the requisite time, and, the nitration being complete, the centrifugal is set in motion and the acid wrung out. In other words, the removal of the nitrocotton and nitrating acids from the pots into the centrifugal machines is avoided. Latterly Messrs. Selwig and Lange have introduced a system in which the centrifugal machine is revolved at a slow speed during the nitration. The acid is thereby forced upwards through the hollow boss of the basket and comes out again in a number of small streams at the top, thus continually circulating. I am perhaps a heretic, but I have never been able to see the advantage of these nitrating centrifugal machines. They cost a great deal of money; they are liable to get out of order; one can only nitrate about 8 kgs. of cotton in each, and with a nitrating period of, say, half-an-hour, one can at the best make ten charges a day in each; further, if the nitrating time is an hour, the number of nitrations is about seven only. This means that for a fairly large production one requires a large number of centrifugals, and it is easy to calculate what this would mean in an artificial silk factory producing, say, three tons of nitrocellulose per day. The quantity of acid used for nitration must be greater, because the space between the basket containing the cotton and the jacket of the machine has to be filled up with acid, and similarly there are a good many other disadvantages. There is no difficulty in arranging pots or basins in such a way that the fumes arising from them are led away by means of an

FIG 3.—NITRATING CENTRIFUGALS

[To face p 32

earthenware fan into an absorbing tower, just as is done in nitrating centrifugals and discharging these nitrating vessels into a wringing machine without its being necessary to expose the workmen to fumes or spilt acid. Such factories have been working very many years and give every satisfaction. In artificial silk factories, where speed is of the greatest importance, the acid is eliminated by putting the nitrated cotton into a lead basket and applying hydraulic pressure on a piston. At the same time such factories do not trouble much about the analysis and the exact conveyance of acids, but all the acid running out from the hydraulic press is conveyed in open cast-iron gutters into a large storage tank, into which revivifying acid runs in continuously at a definite rate, thus keeping up the average of the acid throughout the day. I know of a factory where for the last six years the acid has never been renewed, but only revivified in this way.

Since there is an excess of waste acid produced in revivification, this waste acid is sometimes denitrated in the same way as the acid from nitroglycerine manufacture, but may more advantageously be used in manufacturing fresh nitric acid, because in this case the nitric acid contained in the waste acid is recovered as pure monohydrate.

Revivification is now-a-days very frequently carried out with sulphuric acid containing 20 per cent. of sulphuric anhydride (oleum). This reduces the excess of waste acid very considerably, but, as I have said, it ought to be ascertained whether the resulting nitro-cotton is not affected thereby.

The immersion of the gun-cotton removed from the centrifugals is chiefly carried out by direct dipping into water, but one factory has conveyed it for a long time in a lead gutter along with a large quantity of water, whilst a similar drowning apparatus is largely introduced by Messrs. Selwig and Lange, after having been tried in Waltham Abbey and given up on account of its alleged danger.

When the gun-cotton is pulped and finished it is frequently packed and pressed into boxes. Gun-cotton can become mouldy on the outside through fungi, and, according to v. Forster, have its structure destroyed;[1] and v. Forster found this was promoted by paper in the cases, whilst Malenkowicz[2] showed this to be due to moisture acting on the wood of the boxes. It is very important to

[1] Max v. Förster, "Versuche mit comprimirter Schiessbaumwolle," Berlin, 1883, p. 11.

[2] "Mittheilungen uber Gegenstande des Artilleriewesens," 1907, p. 599.

select proper packing material, on account of the possibility of detrimentally influencing the stability.

A new process for the nitration of cotton is due to Messrs. James Milne Thomson and William Thomson of Waltham Abbey,[1] and it has already been introduced in some factories. An earthenware funnel-shaped vessel can be connected at its stem by means of cocks, either with a pipe supplying fresh acid or with a discharge-pipe An earthenware grating closes the opening of the stem, the new acid is introduced, the cotton dipped in it in the usual way, and then segments made of perforated earthenware plates are laid on top so as to immerse the cotton completely. A small vessel with four outlets is now laid on top, and a Segner wheel distributes water evenly into it, and this is so regulated as to flow out quite slowly and lay itself on the top of the acid without disturbing the latter. This layer of water retains all fumes that may arise from the acid, so that the air in the room is quite good. When nitration is finished water is again allowed to run in, but at the same time connection is made with the outlet-pipe, and the flow of the water being carefully regulated, it gradually displaces the acid. Finally the nitrocotton can be given a preliminary washing.

The advertisements of the makers of the apparatus state that only 0 4 per cent. of acid are lost, and that the acid recovered is as to 70 per cent. with 9·7 per cent of water, and as to 30 per cent. with 20 per cent. of water. With the ordinary processes practically an equal amount of acid is retained by the gun-cotton; therefore when using, say, 30 acid to 1 cotton, about 3·5 per cent. of waste acid are lost; on the other hand, there is no weak acid produced, but all has 10 per cent. of water only. This process gives very good results, and is very convenient for making gun-cotton as required for the British Government, which contains a fairly large percentage of soluble nitrocellulose. As yet there are hardly sufficient data available to decide whether the displacement process will give equally good results for gun-cotton with a small percentage of soluble, or, what is far more important for smokeless powder, whether it will enable a soluble nitrocellulose with definite properties to be made, which, as is known, is always a somewhat difficult matter

The only improvement made in the working up of finished gun-cotton has been patented by Hollings,[2] and further improved

[1] British patent, No. 8278 of 1903.

[2] *Id*, No. 19806 of 1898 and No 28449 of 1899.

FIG 4.—NITRATION OF COTTON (THOMSON'S PATENT DISPLACEMENT PROCESS)

by others. It is a special method of compression, whereby large charges, such as for shells, torpedoes, etc, can be made in one piece, instead of having to be composed from a number of segments, and then turned on a lathe.

Of other modifications of cellulose very little use has so far been made. There is no need for me to dwell upon the defects of nitrosugar and nitromannite, etc., nitro-starch, which is so tempting, has often been tried, but it was impossible to make it stable, besides, nitrostarch could not be nitrated so highly. It was therefore somewhat of a surprise when Arthur Hough of New York[1] announced that he could nitrate starch so as to contain at least 16 per cent. of nitrogen. He did this by dissolving the starch in nitric acid at a temperature of 90° F., and precipitating the nitrostarch by passing gaseous sulphuric anhydride through the solution. In a later patent he altered his process by nitrating the starch with a mixture of 3 parts of nitric acid of 95 per cent. monohydrate and 2 parts of 98 per cent. sulphuric acid, and adding so much sulphuric anhydride to it as to have a concentration of 100 per cent with 1 to 2 per cent. of free SO_3 in the solution. Further, during nitration he injects more sulphuric acid having an excess of about 2 per cent. anhydride into the mixture, and in this way he obtains nitrostarch, which is almost wholly a so-called octonitrate $[C_{12}H_{12}(NO_2)_8O_{10}]$, and contains about 16·5 per cent. of nitrogen. You will remember that Hoitsema[2] has already studied the possibility of producing higher cellulose nitrates than hexanitrocellulose, by keeping up the strength of the acid with phosphoric anhydride; Hough seems to have found the practical solution. This nitrostarch has been utilized in the manufacture of smokeless powders, and I understand that it is used to a certain extent in the United States Army.

[1] British patent, No 12627 of 1904

[2] "Zeitschrift fur angewandte Chemie," 1898, p. 273.

III

In the year 1580 Michel Eyquem de Montaigne, in his "Essais," wrote with reference to gunpowder: "Except to astonish the ears, to which by now everybody is accustomed, I believe this is a weapon of very little effect, and I hope that we shall one day give up its use."[1] Would anybody have dared to repeat such a thought thirty years ago? Yet it has come true.

About the year 1410 we find that quaint treatise on gunpowder called "Feuerwerksbuch," said to have been written by a master gunner, Abraham von Memmingen. It contains the famous history of how Berthold Schwarz tried to make a gold paint and invented gunpowder and guns instead. This book was lent to other master gunners, who severally copied and enlarged it, until in 1534 it was printed in Frankfort-on-the-Main under the title, "Buchsenmeysterei." In this printed edition we find a prescription, "how to shoot out of a gun as far with water as with gunpowder." Take 6 parts of nitric acid, 2 parts of sulphuric acid, 3 parts of liquid ammonia, and 2 parts of "oleum benedictum" (crude tar oil), and charge the gun to a tenth part of its bore. It further advises quaintly, "Light it quickly, so as to get away in time. See that the gun is very strong. With an ordinary gun you can shoot 3000 paces with this water, but it is splendid." This is the first evidence of a nitrated organic substance having been used as a propellant.

I have already alluded to the history of the invention of gun-cotton, but one reference remains to be given, showing how early the use of gun-cotton in rifles was thought of. It is known that Schonbein reported on his gun-cotton on March 11, 1846, and on May 27, 1846, he made experiments with rifles. Professor Otto of Brunswick had, independently of Schönbein, also made gun-cotton, and published his results on October 5, 1846. He also tried gun-cotton in a rifle, and Dr. Hartig,

[1] "Sauf l'étonnement des aureilles, à quoy désormais chascun est apprivoisé, je crois que c'est une arme de fort peu d'effect et espère que nous en quittons un jour l'usage."

a Councillor of Forestry, together with Chief Forester von Schwarzkoppen, certified to have been present when experiments with a rifle and ball-loading were made. This same Dr. Hartig published a pamphlet in 1847 at Brunswick, under the title, "Untersuchungen uber den Bestand und die Wirkungen der explosiven Baumwolle" (Experiments on the Condition and Effects of Explosive Cotton), and therein he makes a statement, which has since attained great importance. He says that the effect which acetic ether has on "the shooting fibre" is very remarkable. He has found that if he makes a stiff, clear jelly with this ether from the shooting fibre, it does not alter its chemical state, and if put in a thin layer on a plate of glass, a snow-white residue is left after the ether has evaporated. If this residue is put into dilute alcohol and then dried it will have in every respect the same properties as the shooting fibre. He mentions already that probably on account of the altered state of aggregation there is a considerable diminution of the explosive force.

Nothing was heard of a real powder made of nitrocellulose for a very long time. It is true that in 1847 the "Commission de Pyroxyle," which was appointed in France, "experimented with it in every form, as wadding, spun, twisted, woven, *reduced to powder by the action of paper-makers' cylinders*, felted together by means of dextrine, finally *granulated like cannon powder*," [1] but it was too violent for use in guns and rifles. Baron von Lenck, in Austria, made gun-charges from fibrous gun-cotton, and we know that they were not a success. In 1865 Captain Eduard Schultze of Berlin published a pamphlet on his "new chemical gunpowder," in which he gave the first indication of his powder, but more words than details. At the same time, however, a number of German journals published some particulars of its manufacture. According to them it was made from wood, which was formed into veneer, and a punching machine punched small discs therefrom. These wood discs were boiled with soda and washed, after that boiled with steam, again washed for twenty-four hours, then bleached with bleaching-powder, washed again and, after drying, nitrated in about the same way as collodion cotton is nitrated now-a-days. After washing, the nitrated wood was boiled in soda solution, and, after further washing, soaked in a saturated solution of potassium nitrate and barium nitrate. Very soon after, Schultze used finely-pulped nitrocellulose, and made powder grains by agglomeration with

[1] "Note sur la pyroxyline ou coton-poudre," par M. Susane, Mémoires de l'Académie Impériale de Metz, 1855.

water in drums. It is also remarkable that in 1865 Abel[1] patented the production of grains of gun-cotton by placing a mixture of gun-cotton with water and a little gum-arabic in a pan, and giving it a shaking motion, whereby the gun-cotton was formed into grains He also proposed to mix soluble and insoluble gun-cotton, and to make the soluble gun-cotton serve as a binding material by treatment with wood spirit, alcohol, ether, or mixtures of these liquids It is further interesting that Dr Kellner of Woolwich is mentioned in a German book which appeared in 1866[2] to have been the first to succeed in making a granular smokeless powder. Neither Abel nor Kellner seem to have continued at the gelatinization of nitrocellulose

The author well remembers, however, a firm in Marchegg, near Vienna, which existed under the name of Volkmann's k k. priv. Collodinfabriks Gesellschaft H Pernice & Co. They originally bought the patent for the Schultze powder, and made it under the name of Nitroxylin. From 1872 to 1875 they made a powder called Collodin, the invention of Friedrich Volkmann, which was patented under date November 8, 1870, and May 31, 1871. After three years of existence the Austrian Government ordered the works to be closed, because they claimed that this explosive was infringing their gunpowder monopoly. Thus this powder was lost to the world, and no information regarding it was procurable until recently, when the author succeeded in obtaining a copy of these most remarkable patents (A literal translation is to be found in the Appendix.)

Volkmann cut up alder-wood into small grains of the size of black powder He boiled them in a 3 per cent. soda solution, washed them, treated them with steam, and washed them again. He then bleached them in a solution of bleaching-powder, and after final boiling with pure water, nitrated them in a mixture of nitric and sulphuric acid. Thus far the treatment was that usually given to cotton-waste. The finished grains were soaked in a solution of potassium nitrate, or of potassium nitrate and barium nitrate, and, after drying, treated with a mixture of 5 volumes of ether to 1 volume of alcohol. This was carried out in two different ways The grains were either covered with ether-alcohol, left therein for between 3 and 30 minutes, according to size, and taken out before the solvent had penetrated right through. They were then agitated by means of air, and also sprinkled with dry powder

[1] British patent, No. 1102 of 1865.
[2] "Buch der Erfindungen," Leipsic, 1866, chapter on gunpowder and arms.

dust to prevent their sticking together, and subsequently dried. Alternatively the solvent was allowed to penetrate the grains completely, and the more the substance was dissolved the more the volume decreased. On taking the powder out of the solvent it had the appearance of a mush, which, after 12 hours' drying at 30° C., was converted into a dough, a pasty, pliable substance, from which any shape could be obtained by moulding and pressing. By dissolving and pressing to a greater or lesser extent the rapidity of combustion in rifles of these shaped bodies could be modified at will He claimed that "the advantages of this powder over the chemical wood powder consist in its vapour being so transparent as not to prevent the eye from seeing the object on shooting in rapid succession, in having a lesser report, in leaving little dry, ash-like residue, which is washed away by the succeeding shot, in requiring half the weight of black powder in order to carry the projectile one-third further with a quarter greater muzzle velocity; in having one-half straighter a trajectory; in having an uniform effect, inasmuch as it is not a mechanical mixture; in its manufacture, storage, and carriage being unattended by danger, in its being undamaged by moisture; in its volume being considerably reduced in relation to its density, whereby it can be used even for rifles having a very small chamber for the charge, and in its being more economical for blasting purposes Truly Volkmann seems to have known everything about a smokeless powder.

Patents at that time were kept secret in Austria, and thus his method of manufacturing this powder was not known to the world at large.

In 1882 Mr Walter F. Reid patented[1] the agglomeration of nitrocellulose into grains and moistening them with ether-alcohol for the purpose of hardening the grains. I had the advantage of seeing this manufacture and some experiments with this powder in 1883, in which year also Oscar Wolff and Max von Forster published and patented[2] the method of coating small cubes of gun-cotton with a solvent for the purpose of keeping them permanently moist. Mr. Reid's powder is manufactured under the name of E C. powder, and is still a favourite sporting powder, but being what is now called a bulk powder, namely, a powder of very loose structure and low volumetric density, it was too violent in its effects for military rifles, whilst for sporting rifles it was just the right thing. I would again mention here that in the beginning of 1886 I suggested to Professor Hebler, the well-known Swiss

[1] British patent, No. 619 of 1882. [2] *Id.*, No 3866 of 1883

pioneer of the small-bore rifle, the use of a piece of blasting gelatine as a charge for a rifle cartridge,[1] but that the very idea frightened him, although he wished to have a pellet of compressed gun-cotton from me for the purpose. Vieille in 1886 [2] thoroughly gelatinized nitrocellulose, and made sheets of it, which he cut up in strips or small lozenge-like squares. This was the first military smokeless powder. It has been said that Vieille made his discovery whilst trying to make a bulk powder similar to E C powder, but I have it from him that his invention was the outcome of prolonged study and experiment to determine the effect of increased loading density on the rate of combustion, leading him to gradually increase the solvent action until he obtained a thoroughly gelatinized powder.

This impartial survey shows that whilst the merit of making the first powder-like material from a nitro-compound belongs to Hartig, and whilst Schultze made the first commercial powder, yet the invention of a gelatinized powder in the modern sense must be attributed to Friedrich Volkmann, although independently of him Reid rediscovered, twelve years later, a hardened sporting powder, and Vieille, sixteen years later, a thoroughly gelatinized military powder. It appears, therefore, that the Austrians were not only the first to experiment with gun-cotton in guns, but also had the present-day smokeless powder before others, only to crush it out of existence by their monopoly. The clock of industrial progress was thereby virtually put back fifteen years

The division of smokeless powders into such that are made of nitrocellulose alone and such that are made with nitrocellulose and some other nitro-compound, might now-a-days well be reduced to nitrocellulose powders and nitrocellulose-nitroglycerine powders. All other smokeless powders had a comparatively short life, and were not introduced into the service of any army. Nitroglycerine-nitrocellulose powder was invented by Alfred Nobel in 1888,[3] who gave it the name of Ballistite. The British Government adopted a powder which contained insoluble gun-cotton with nitroglycerine and vaseline, the whole being dissolved in acetone.[4] Nobel at first used for his Ballistite soluble nitrocellulose with nitroglycerine and camphor, but the latter ingredient was afterwards discarded. Ballistite is the

[1] "Journal of the Society of Chemical Industry," 1894, p. 575.
[2] "Mémorial des Poudres et Salpêtres," 1890, p 9.
[3] British patent, No. 1471 of 1888. [4] *Id.*, No. 5614 of 1889

Service powder in Italy, and is much used for large guns Aniline is now added, and it is claimed both for vaseline, aniline and diphenylamine that they exert a great stabilizing influence on the powder.

There is no need for me to detail the manufacture of powders. This is a subject of great mechanical interest, and although the shape and the physical condition of the powder have a considerable influence on its shooting qualities, yet it is not intended to include the machinery in the present Lectures Nitrocellulose powders are made from dry nitrocellulose, in a mixing machine, using a solvent (generally ether-alcohol or ether-acetone). In many factories the nitrocellulose is made anhydrous by soaking it in alcohol, first of all in such that has already been used for this purpose, and then in pure alcohol, squeezing it out every time in a press. Some think that the quality of the powder is affected by this method, whilst it is more probable that certain unstable compounds, or such that could affect the regularity of shooting, would be eliminated by the alcohol treatment. Some have found a loss of substance up to 5 per cent. to take place with certain nitrocottons. The mixture is then rolled under a pair of heavy rolls into sheets of the required thickness, cutting them up into squares, and afterwards drying these. In some Services ribbons are used instead of grains, and in others threads or tubes are made of such powder and cut into sticks of the length required for the charge. In Germany camphor was formerly added to the powder whilst kneading it. Some countries leave a certain amount of the solvent in the powder, and formerly in France a little amyl alcohol was added, while now diphenylamine has been adopted, this was already used in 1889 in Germany for C/89 powder made by the Cologne-Rottweil factory. The threads of powder, generally squeezed out of a die by hydraulic pressure, are frequently hung up on clips in drying cupboards, or else cut into sticks and dried on trays, thus giving a good opportunity for recovering the solvent. Flake powder and stick powder are frequently boiled in water to free them from solvent; nitroglycerine powders, like Cordite, undergo the same treatment as pure nitrocellulose powders.

Ballistite is made in a different way. Soluble nitrocellulose in the shape of a fine powder is suspended in fifteen times its own bulk of water and nitroglycerine added, the mixture being stirred by means of compressed air. This causes the nitroglycerine to dissolve up the nitrocellulose, the water acting as a carrier only.

This process is the invention of Messrs. Lundholm and Sayers,[1] and has enabled as much as equal weights of nitroglycerine and nitrocellulose to be incorporated. The paste resulting in this way is freed from water in a centrifugal machine and allowed to ripen. It is then brought under steam-heated rolls, weighted to exert a pressure of 100 atmospheres, to thoroughly incorporate it, and then mixed by rolling the sheets over and over until they are quite satisfactory. The sheets so obtained are then cut up into flakes, cubes, strips, etc., as required. Another powder made in Italy is Solenite, which consists of 30 parts of nitroglycerine, 40 parts of insoluble and 30 parts of soluble nitrocellulose, the two having an average of 12.6 per cent. nitrogen. Acetone is added for promoting the solution of the insoluble nitrocellulose. This is an outline of the manufacture only, and it would not be right to give further details here.

I ought to say a good deal regarding the various forms in which powders are made now-a-days, but I could hardly do that without going more fully into the machinery required to give them these forms. You will readily understand that every weapon may, and generally does, require a different powder in order to give the desired velocity and not to exceed the permissible limits of pressure. It is obvious that it would be very easy to alter the composition in every case, but as a matter of course such an expedient would be quite undesirable alike from a manufacturing and from a Service point of view. Hence, already in the days of black powder it has been the custom to vary the shape and size of the powder. We thus have ribbons in France, strings in Great Britain, flakes and tubes in Germany, cords of square section in Italy, short multiperforated cylinders in the United States, cubes from Ballistite, spiral sporting powder in Germany, the *poudre peigne* (spiral powder with comb-shaped incisions) of French inventors, etc. Further, these powders may then be made in various lengths, breadths or thicknesses, and with various kinds of holes, incisions, etc. It is quite impossible to generalize and to say that a particular form is good or bad, because it probably does suit a special weapon. It is a fact that up to a certain size, round grains are most likely to give good combustion, and that cord or tube comes next; on the other hand, a flat ribbon is likely to burn more uniformly, although again a variation in the rate of combustion at different intervals of time may just be what is wanted.

The conviction has grown of late that in addition to being smoke-

[1] British patent, No. 10376 of 1889.

less a powder should also be flameless, so as not to disclose the position of an attacking force Smokeless powder does show a very strong, luminous flame, chiefly on account of its high temperature of explosion, which causes the particles of residue to become incandescent, and also owing to the fact that some of the powder charge always escapes combustion in the gun and follows the projectile as a luminous tail for a short distance The problem being somewhat similar to that presented by explosives in coal mines, it is not surprising that one of the first patents in this connection, that of Duttenhofer,[1] proposes to add sodium bicarbonate to the powder, a substance which has the effect of cooling the flame by losing its water of crystallization and carbon dioxide. Other substances, like oils, soaps, etc., are employed, but the matter is not yet in a sufficiently advanced state to permit of an opinion being expressed.

Various circumstances underlying the manufacture of these smokeless powders combine to affect their quality The military powders suffer, in the first instance, from irregular shooting This is, of course, due to defective composition, be it *ab initio* on account of the selection of unsuitable proportions, or of bad or careless manufacture. With military powders the composition and methods of manufacture are generally laid down in accordance with experience and tests, but this introduces the danger of too narrow limits, with the result that everything goes wrong, when there is the slightest variation in, say, the nitrocellulose or the thickness of the powder. In the case of sporting powder, it is necessary to carry out shooting tests with every small batch, because the reputation of a firm depends on keeping powder out of the market which is in the slightest degree deficient Careful blending has to be resorted to in order to obtain absolutely uniform results throughout.

Of other difficulties in manufacture I will mention only a few. The treatment of a powder under rolls is to a certain extent guided by rule of thumb It is all very well to look through the paste the sheet may appear quite transparent to a good and experienced eye, yet small nodules of nitrocellulose may have escaped solution for a long time The constant crackling heard when rolling thin sheets plainly points to such isolated and undissolved fibres Incorporating in a kneading machine does not improve matters more especially as these machines are really poor mixers, although some knead very well. Pressing powder out of a die gives

[1] British patents, No 19408 of 1906, and No. 791 of 1907.

very good results with small diameters, but with larger diameters very much depends upon the shape of, and the wear on the nozzle, its position among several others or relatively to the die, and on whether the outer skin will contain air bubbles or be cracked. If too much solvent is taken, or the proportions of a composite solvent are not quite suitable, the density and uniformity of the powder will suffer. One of the greatest difficulties lies in the proper drying of a powder. The smaller sizes of sticks, ribbons, tubes, etc., are easier to deal with, although they also would really require more attention than to simply leave them for a certain length of time in a stove. The larger and thicker ones, however, sometimes require months to dry properly, and it may then be found that the outside of, say, a half-inch cylinder is full of fine hair-cracks, whilst the inside is still comparatively soft. With some powders this defect is to a certain extent avoided by leaving some of the solvent behind, but then, of course, we have on the one hand the difficulty of not knowing exactly when the correct amount of solvent is present, and, on the other hand, a certain amount of risk in that the powder would in course of time undergo changes by gradual evaporation of the solvent.

I have already stated that as far as military powders are concerned there are now-a-days only two varieties, pure nitrocellulose powders and those made of nitrocellulose and nitroglycerine. Formerly all kinds of powders were made, some of very fanciful mixture, but it was found that complicated compositions were most difficult to regulate, and a return was therefore made to more simple combinations. I need not enter into these, every book and the "Patent Journal" will satisfy the curious. Sporting powders are of two kinds, the so-called bulk powders, consisting of loose granules, coated or hardened by means of a solvent, and the so-called condensed powders, gelatinized throughout, and made in practically the same way as military flake powders.[1] The former are supposed to just fill a cartridge used in the old black-powder gun, the latter are made for modern weapons. Another kind of sporting powder, which occupies an intermediate position, is made on the lines of the Walsrode powder, one of the earlier explosives, which still holds the market. This powder is thoroughly gelatinized, but then treated with water or steam, whereby granules are formed, and part of the solvent is driven out again, leaving a "bulky" but hard-grained powder behind. The usual "bulk" powders are composed of

[1] The micro-photographs Figs 5, 6, 7 and 8 were kindly prepared by Mr. Henry de Mosenthal.

FIG 5.—SMOKELESS BULK POWDER SEEN UNDER THE MICROSCOPE IN ORDINARY LIGHT

FIG. 6.—SMOKELESS BULK POWDER SEEN UNDER THE MICROSCOPE IN POLARIZED LIGHT

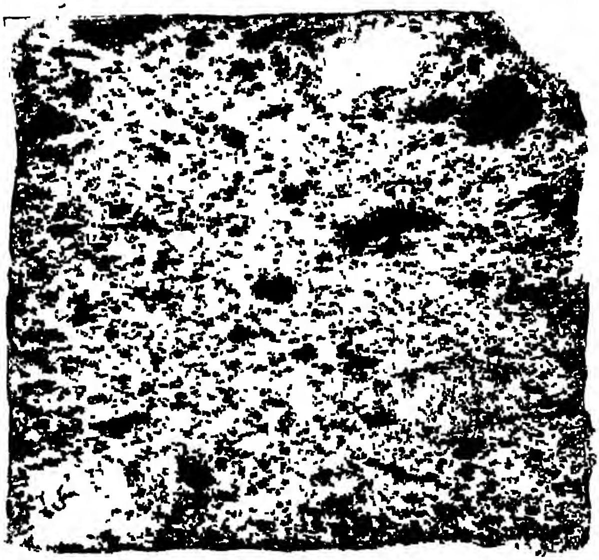

FIG. 7.—SMOKELESS FLAKE POWDER SEEN UNDER THE MICROSCOPE IN ORDINARY LIGHT

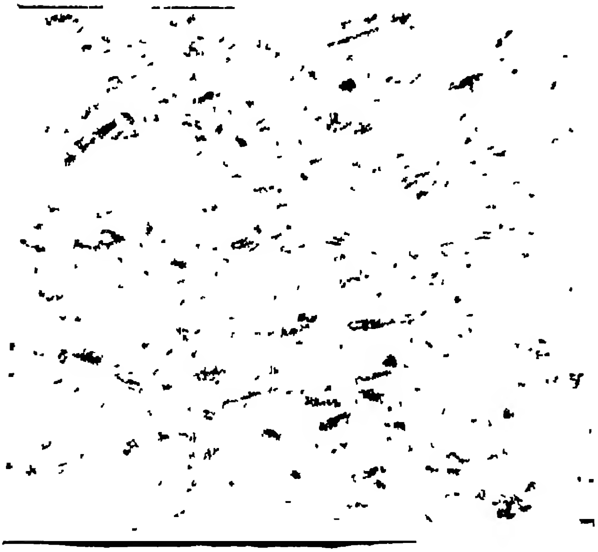

FIG. 8.—SMOKELESS FLAKE POWDER SEEN UNDER THE MICROSCOPE IN POLARIZED LIGHT

[*To face p.* 44

soluble nitrocotton mixed with potassium or barium nitrate, and generally worked up in an incorporating mill or drum. The mixture is then either sprinkled with water in a rotating drum, so as to form grains, or extended on a shaking table making short and rapid oscillations. Alternatively it may be put into an hydraulic press and then broken into grains, the solvent being in every case sprinkled over when the grains are already formed.

The "condensed" powders are usually made by rolling the "paste" into very thin sheets (0 1 mm and less), which are then cut into small flakes to obtain the requisite rapidity of combustion. Such powders are dried fairly quickly, and they may sometimes even be boiled in water to promote elimination of the solvent

The properties, keeping qualities, advantages and defects of smokeless powders and explosives generally I will refer to in my last Lecture.

Since 1800, when Howard invented fulminate of mercury, and since 1815, when Durs Egg made the first cap, but little progress has been made in the manufacture of these articles. It is still the usual cap and the usual detonator, the only difference being that potassium chlorate enters partly into the composition of detonators, whilst for smokeless powders a hotter flame is found essential, and is obtained by adding a combustible substance. The idea of metallic powders also had an influence upon cap compositions, aluminium powder, either mixed with the fulminate or pressed in a layer on top of it, having been successfully employed by Dr. Brownsdon and the King's Norton Metal Company [1] Quite recently Wohler and Matter made fulminates the subject of an interesting piece of research,[2] and found that small quantities of silver nitride suffice to replace the fulminate. Bielefeldt found already in 1900[3] that a small quantity of fulminate placed on top of trinitrotoluene or other aromatic nitro-compound forms an excellent detonating mixture, and the Rheinisch-Westfälische Gesellschaft of Troisdorf make now detonators of Tetranitromethylaniline (called Tetryl).[4] It is said that quite half of all the detonators at present manufactured in Germany are made with trinitrotoluene Hyronimus[5] proposes lead nitride $[(N_3)_2Pb]$ as a substitute for mercury fulminate.

The manufacture of fulminate of mercury is performed in almost

[1] British patent, No 23366 of 1904.

[2] Zeitschrift fur das gesamte Schiess- und Sprengstoffwesen, 1907, p 181. Also British patent, No 4468 of 1908

[3] British patent, No 20133 of 1900

[4] *Id.*, No. 13340 of 1905

[5] *Id*, No 1819 of 1908

the same way as that described fifty years ago. Its colour has, however, been improved from grey to white by the addition of a little hydrochloric acid and copper to the acid mixture, and the alcohol is now recovered from the carefully condensed vapours. The mixing of cap composition has to be carried out on a much larger scale than formerly. Two kinds of mixers are used, both practically identical, inasmuch as mixing is effected by the gentle motion of india-rubber bodies, and both very safe to handle. The English mixer is called the jelly-bag mixer, on account of a silk bag containing india-rubber rings strung up, the German mixer is saucer-shaped and contains india-rubber balls.

The increasing demand for ammonium nitrate safety explosives has resulted in the use of greater quantities of powerful detonators. In the days of kieselguhr dynamite a No. 3 detonator was usual, and a so-called "double force" (No 5) detonator was quite a luxury Now-a-days nearly all safety explosives require a No. 6 detonator, and most manufacturers would be only too glad if the miner could be persuaded to use a No. 8 (2 gram) detonator, so as to make quite sure of perfect explosion. For the same reason great progress has been made with electric detonators. Formerly high-tension fuses fired by frictional electric machines were almost solely used, and Breguets were the only low-tension fuses employed in mines. Now-a-days the tendency is to use low-tension fuses and magneto-firing apparatus, thus greatly reducing the risk of firing the pit gases. Last year the Fabrik Elektrischer Zunder in Cologne tried a system of shot-firing from the surface Formerly only a comparatively short length of wire could be laid without unduly increasing the strength of current required, and the man firing the shot had to take shelter in the mine. This firm introduces *relais* at various intervals, which keep up the strength of the current, so that a permanent main cable can be laid right up to the pit's mouth. When everybody has left the mine the shots are fired, and ample time is thus given for any hang-fires to go off.

Bickford's invention still holds the field as regards safety fuses. I have explained in my first Lecture wherein the few improvements consist that were made on safety fuses. It is curious that all attempts to make a safety fuse with a core of smokeless powder or some other nitro-compound have so far been unsatisfactory. It seems impossible to ensure uninterrupted burning. The Westfälisch Anhaltische Gesellschaft some years ago made a fuse of this kind, which was supposed not to fire pit gas,[1] and was intended

[1] British patent, No 2225 of 1898.

for use in fiery mines. It was, however, evidently in its initial stage when I tested it, and I have not heard of it since. Of late rapid-burning fuses were introduced, some being fired in groups by means of pistols and other central-firing arrangements General Lauer and Mr. Tirmann introduced friction fuses, which are fired by means of wires from a distance, and are extensively used chiefly in Austrian coal mines. Girard made *cordeaux détonants* by filling lead tubes with nitrohydrocellulose and then drawing them out to the diameter of an ordinary safety fuse In 1906 these fuses were filled with Melinite, and now Trinitrotoluene is also used, which permits the employment of lead tubes instead of the costly tin tubes indispensable with a picric acid explosive.[1] The most perfect fuse of this kind is, however, the instantaneous fuse invented by General Hess and introduced into the Austro-Hungarian Service Originally it consisted of a mercuric fulminate core on four threads. In 1903 Hess "phlegmatized" the fulminate [2] by the addition of 20 per cent. of hard paraffin, but a number of such fuses, tied together by knots, can be detonated by a common detonator, thus replacing electric shot-firing and dispensing with a detonator in each bore-hole. The fuse can be cut, hammered, squeezed, etc., without danger. More than thirteen years ago I could not understand why private manufacturers had not commenced to manufacture Hess's fuse for use in mines, and more especially for safety explosives, for which purpose they are readily adaptable. The "phlegmatized" fuse is a great improvement, and I can therefore emphatically repeat my recommendation.

The more industry progressed all over the world, the greater the coal consumption became, and the more frequently occurred those appalling mine disasters which from time to time convulse public feeling. The British Government was the first to nominate a Fire-damp Commission, but this only recommended a palliative measure in the shape of a water cartridge. Then followed commissions in Prussia, France, Saxony and Austria, but not one of them tried a safety explosive before September 1885 The Prussian Commission at their meeting on June 25, 1885, permitted by 13 votes to 10 the use of dynamite where less than 3 per cent. of gas was present. The Commission even failed to ignite air containing between 3 and 10 per cent. of pit gas by means of gun-cotton. Tonite was found unsafe; compressed

[1] Artilleristische Monatshefte, August 1908.

[2] "Mittheilungen uber Gegenstande des Artillerie und Geniewesens" 1907, p. 115

Schultze powder was safe in a 10 per cent. gas mixture, but it was considered liable to decompose and to be very hygroscopic. The Prussian Government, however, had built a testing station and trial gallery at Neunkirchen, in the beginning of September 1885, under the direction of Mr. Margraf, who tested Hellhoffite, made by Messrs. Schmidt and Bichel, and consisting of nitric acid and nitrobenzene. Although this proved safe in the usual pit gas mixtures, yet it was found impossible to manipulate it, so another explosive, "Carbonite," made by the same firm, was tried. This was safe in small charges only. Improvements were therefore made, and in September 1887 a Carbonite consisting of saltpetre, cellulose, nitroglycerine and sulphuretted oil was found to be absolutely safe. In 1886 Margraf tested Securite (dinitronaphthalene and ammonium nitrate mixed with an alcoholic solution of nitrated resin) against Carbonite, and this also was found safe. In April 1887 Roburite and Kinetite were tried, and in August 1887 soda dynamite. Thus Carbonite was really the first safety explosive, and curiously enough it has not yet been surpassed for safety.

It is necessary to distinguish between explosives which are safe in manipulation (handhabungssicher) and such that are safe in fire-damp (wettersicher). The latter only are called safety explosives in this country, and I cannot understand why it was resolved at the Congress of Applied Chemistry in Rome to apply the words "safety explosive" to those safe in manipulation only, when twenty-five years' usage clearly pointed to another meaning. At any rate even those who proposed this resolution have since been unable to bring it into effect.

The obvious question is: What makes an explosive safe in fire-damp? I confess that, having most carefully examined the views of those most competent to give an opinion, I fail to find a definite answer At one time the Prussian Commission stated that the more rapid the explosion the safer the explosive, and some colour is lent to this theory by the fact that fulminate of mercury does not ordinarily ignite fire-damp, whilst black powder always does. The theory is, however, controverted by certain black powder mixtures, foremost among which is Bobbinite, which is safe up to a certain point, and by nitroglycerine and blasting gelatine, which are not.

The French Government Commission stated that a mixture of methane and air would ignite at 650° C, but that ignition was delayed for about 10 seconds, and therefore a much higher temperature was necessary, so that an explosive, whose temperature

of explosion, as calculated by thermochemical data, was below 1500° C. could be licensed for use in fiery mines. Curiously enough Carbonite, so far the safest of all, and several others, which are licensed for such use, have a temperature of explosion considerably higher than 1500° C.

Mr Bichel, to whom in conjunction with his collaborator, Dr. Mettegang, we owe most excellent methods for examining explosives, says that the velocity of detonation, the maximum temperature of the products of combustion, the length and the duration of the flame of an explosive all influence the safety of an explosive adversely.[1] Whilst this is undoubtedly correct, he has thereby abandoned the theories of others, and this perhaps advantageously. Thus he requires a slow detonating explosive, and does not trouble about the temperature of explosion, but about the temperature of the products The length and duration of the flame are evidently a function of the rapidity of explosion and the quantity of its products. He therefore considers, and in the author's opinion very justly, the nature of the products of combustion to be all important, whether they consist of solid particles which remain incandescent for a considerable time, or of large quantities of combustible gases shot forward with great force. In this way he corroborates early attempts to photograph the flame of an explosion made by Schoeneweg, the inventor of Securite, and by Siersch of Pozsony. He does this by placing the whole matter on a scientific basis, using ingenious apparatus for determining each factor. The velocity of detonation cannot, however, be considered to be a determining factor under all circumstances. Certain nitroglycerine explosives, amongst which we may also include Carbonite, explode much more rapidly than, say, Bobbinite, and yet show themselves to be much safer when tested. I myself have found that up to a certain point the addition of picric acid gave increased safety on test.

It will be remembered that the British Commission found a water jacket round the charge very efficient. In Austria they followed this up by using wet moss and sand, and thus the idea of using water of crystallization instead was evolved. Sodium carbonate, magnesium sulphate and other substances were tried, either separately in front of the explosive, or as ingredients. More prominence was then given to the French recommendations, and the notion became prevalent that the addition to the explosive must be a flame-cooling agent in the shape of water vapour or some other heat-absorbing gas. Thus permanganate, bichromate

1 "Gluckauf," 1904, No. 35

oxalates and other salts were used, and of late common salt has sprung into favour.

The only definite result obtained so far is that ammonium nitrate is absolutely safe in all quantities, and that cellulose and similar substances in nitroglycerine compositions, *e. g.* rye flour in Carbonites, or wood pulp in other explosives, renders them highly inert in fire-damp mixtures. Ammonium nitrate cannot, however, be used by itself, although Lobry de Bruyn succeeded in exploding it,[1] and therefore some combustible substance must be added. It simply remains to be determined what minimum quantity of such combustible can be added to avoid flames of great length and duration.

The next question is how can one tell whether an explosive is "safe." This question is a still more difficult one to answer. The various Governments and also certain factories have erected testing stations, in which explosives are tested in mixtures of air with natural pit gas, artificially produced methane, coal gas, benzene, etc. I myself have had the privilege of designing a testing station of this kind for a German factory. These stations generally consist of a long wooden or iron tunnel, round or oval in section. The explosive is shot at a definite angle with the roof out of a mortar into a gas mixture, thus imitating the natural conditions in a mine as closely as possible. In some of these stations a short section may be partitioned off for special tests. In this country a ballistic pendulum is used to ascertain the quantity of the explosive equal in force to 4 ounces of dynamite No. 1, and this quantity with a stemmed shot is then fired in air containing 15 per cent. of coal gas. If the mixture does not fire in twenty shots the explosive is considered a safe one. In most other countries the quantity of the explosive in question is determined which will fire and that which will just not fire a certain pit gas mixture. This gives us what Mr Watteyne, the well-known Belgian authority, calls the *charge limite* of an explosive. This latter way is certainly the more rational one, since it permits of comparison between different kinds of explosives. Is this method of testing, however, above reproach? I think not, although I know of no better one at present. It has been found that the narrower the bore of the cannon the easier ignition takes place under certain circumstances. The Woolwich circular section gallery, which has a sectional area of 0·36 square meters, is much more sensitive than the elliptical Belgian one, whose sectional area is 2 square meters, and, in fact, even

[1] "Recueil des travaux chimiques des Pays-Bas," 1891, p. 127

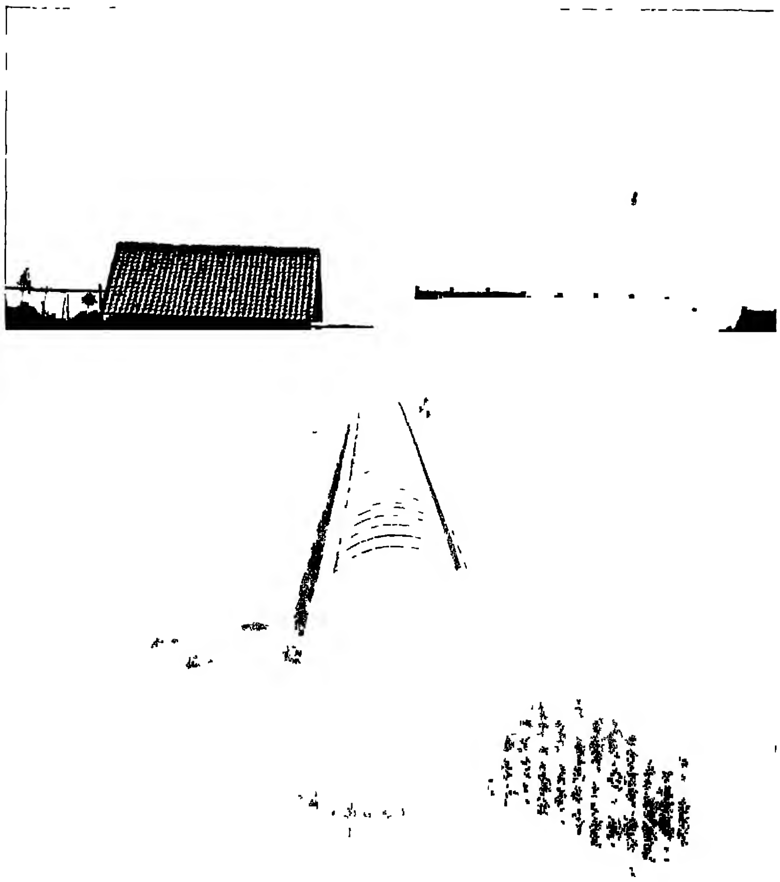

Fig. 9.—TESTING STATION AT FRAMERIES (END VIEW)

[To face p. 60

FIG. 10.—TESTING STATION AT FRAMERIES (VIEW OF THE INTERIOR)

[To face p. 50

with equal diameters each gallery may be said to have its own ignition temperament, which affects the results. Thus quite recent tests at Frameries in a gallery having a sectional area of 0 28 square meters showed that two safety explosives, whose *charge limite* was 900 and 450 grammes respectively, fired at 300 and at 75 grammes. The gas used also exerts considerable influence on the tests, and it is a great pity that the proposal made at the Berlin Congress of Applied Chemistry to carry out comparative tests between certain gas mixtures was not adopted.

It has been known for a long time that coal dust as well as pit gas is highly explosive, and in the early days of safety explosives tests were made in coal dust alone and in coal dust mixed with pit gas I believe that Engler, when investigating explosions in the charcoal heaps of the Black Forest,[1] was the first to show that mixtures of air with coal and other carbonaceous matter, which on heating develops combustible gases, could be exploded, but that this was not the case with charcoal or soot. Mixtures of coal gas and air, however, so poor in gas as to be non-inflammable, were rendered explosive by the addition of some charcoal dust. Other experts, such as Mr. Simon of Liévin,[2] have carried out and described many such experiments, whilst Messrs. Martin and Hall, the British Inspectors of Mines, have preached in vain against dusty roads Ultimately, when mere watering of mine galleries proved to be of little use, the Mining Association of Great Britain took the lead, experimentally investigating the influence of coal dust on explosions in mines. As is usual, anything done in this country is done thoroughly, and an iron shell 7 feet 6 inches in diameter and 1,083 feet long was used to carry out the experiments. So far it has already been ascertained [3] that two zones of stone dust on either side of a zone of coal dust arrested the path of a flame, and that unless the coal dust zone exceeded 180 feet in length, no explosive force was manifested. Might I submit an old idea, which I base on some patents of mine that have proved highly useful? An absorption tower retains solid particles contained in a gas mixture, and also cools the latter very efficiently, and one of the best methods for absorption has proved to be the production of a fine spray or mist of moisture. It seems quite feasible to utilize certain lengths of tunnel for the construction of inverted absorption towers at intervals, and certainly at every point where a side gallery runs into the main or haulage

[1] "Chemische Industrie," 1885, No 6

[2] "Annales des mines," November, 1890.

[3] Coal Dust Experiments, "The Times," September 24, 1908

roads. Water is plentiful in a mine, high-pressure pumps are always available, and it would therefore only be necessary to install and maintain a number of spray nozzles, like those used for absorbing and cooling steam in large generating plants, to permeate an air zone, say, 20 yards long with fine water mist. I think a number of such zones would absolutely prevent the ever-present danger of transmitting an isolated explosion to the whole of the mine, whilst it will cause no inconvenience and may even beneficially affect the miners. I hope this idea will be given a trial in the near future. So much seems certain to me from the study of the results of past investigators that a small addition of coal dust will be found to promote the explosion of poor gas mixtures, and that, therefore, a separation of the dust from the gas will in some cases prevent an explosion.

Lacking definite knowledge as to what renders an explosive safe in fire-damp, and how this is to be ascertained, it would be natural to seek a solution in practical results. The sale of an article does not always depend upon its real value, but very frequently on the way it is advertised and pushed, whether it is made in the country of consumption or not, whether it possesses disadvantages that render another less efficient article a preferable one, etc. In spite of this it is not unfair to assume that the statistics showing the quantities of safety explosives actually consumed in a great coal-producing country like Great Britain have a real bearing on the question as to which explosives have given a reasonable amount of safety. The report of the Inspectors of Explosives for 1907 gives the following highly instructive table. Out of a total consumption of 7,764,122 lbs were used :—

NAME OF EXPLOSIVE.	QUANTITY USED LBS.	PERCENTAGE OF TOTAL
Saxonite	1,721,193	22·18
Bobbinite	1,063,111	13 69
Monobel Powder ...	711,691	9 17
Ammonite	562,405	7 25
Carbonite	551,948	7 11
Roburite ..	510,438	6·57
Arkite	437,780	5·64
Westfalite	405,691	5 22
Bellite	371,455	4·78
Rippite	306,408	3·95
Faversham Powder ...	224,200	2·88
Stowite	180,393	2 32
Ammonal	114,806	1 48

Of these Saxonite, Monobel powder, Carbonite, Arkite, Rippite,

and Stowite contain large percentages of nitroglycerine. Bobbinite is a black powder mixture, the rest are ammonium nitrate explosives.

From the inquiry on Bobbinite[1] the following table regarding accidents in coal mines caused by various safety explosives in 1904 and 1905 is calculated—

	CONSUMPTION.	ACCIDENTS.		KILLED		INJURED	
In 1907	%	NO	%	NO.	%	NO	%
Bobbinite ..	13·69	20	17·54	2	8·33	30	18·87
Other permitted explosives	86·31	94	82·46	22	91·67	129	81·13

It will thus be seen that a black powder mixture like Bobbinite, which would not be licensed in any other country and be condemned without trial, ranks second in consumption, being used to the extent of 13·7 per cent. of the total consumption, whilst Saxonite, a nitroglycerine explosive, ranks first, with 22·18 per cent. of the total.

Am I therefore right in saying that we have succeeded in making the use of explosives in coal mines infinitely more safe than before, but that we do not really know why?

[1] Report of the Departmental Committee on Bobbinite, London, 1907.

IV

There are still a few applications of explosives to be mentioned, and after that we may pass on to more general considerations.

Nitrocellulose has found a greater sphere of use for purposes other than smokeless powder or dynamites. The celluloid industry, introduced by the Brothers Hyatt, and more recently the artificial silk industry, consume enormous quantities Of Celluloid, the United States produce about 4000 tons per annum, Germany 15,000, and the rest of the world about 5000 tons, of which yearly total of 24,000 tons this country produces about 2 per cent. This necessitates about 14,000 tons of nitrocellulose per annum. Of artificial silk, about 5000 tons are made annually, though only about 200 tons in England[1] The amounts used for varnishes like Pegamoid, Fabrikoid, etc., for making or steeping incandescent gas mantles, for waterproofing solutions, for patent leather (nitrocellulose dissolved in amylacetate, and mixed with aniline black) and for photography are also considerable As already mentioned, all these industries have abandoned the use of anything but cotton, because the ultimate product was much more reliable, and at the same time possessed valuable properties, necessarily absent in nitrocellulose made from wood pulp, paper, etc. This is especially the case with artificial silk, for which tenacity and ductility of the spun fibre are as important as viscous, yet easy issue from the orifice of the "silk worm." The solubility of the nitrocellulose in a definite mixture of ether-alcohol to the extent of 2 per cent. either way is by no means unimportant, as this may mean 10 per cent. more of very expensive solvent. When you consider that one of these factories, which I had occasion to revisit quite recently, makes as much as 3000 kgs. of silk a day, you will have some idea of the sums involved.

The nitrocellulose for all these industries should be perfectly

[1] According to Dr. Richard Schwarr, there are at present in Europe 30 factories making artificial silk, and the world's production in 1907 amounted to 3 3 million kilogrammes, of which 1 5 millions were nitrocellulose silk, 1 3 millions "Glanzstoff," and 0 5 millions viscose silk ("Neue Freie Presse, Vienna, January 5, 1909.)

soluble in ether-alcohol, or, in the case of varnishes, in methyl alcohol. Of course perfection cannot be reached industrially, and although this is not so important in the case of celluloid, yet with artificial silk and varnishes special means have to be employed for filtering the solutions clear of undissolved fibre. It is also necessary to avoid turbidity on drying, this being frequently due to moisture.

In neither of these cases is the nitrocellulose pulped, but the whole of the fibre is dissolved. I am afraid purification is sometimes not carried as far as it ought to be with due regard to the stability of the finished celluloid. In the case of artificial silk the fact that the nitrocellulose is denitrated seems to indicate that thorough purification is unnecessary, but the silk fibre made from well-stabilized nitrocellulose will be found to possess inherent good properties of its own The same may be said of varnish, although in this case a slight acidity at certain stages of the process has the advantage of rendering the nitrocellulose more readily soluble. This has also been shown in the investigations of Lunge and Suter.

The manufacture of these nitrocelluloses also varies in other respects. In dealing with such large quantities everything is carried out expeditiously and without much handling Thus the acids are measured and not weighed, which is quite as accurate a method. The nitrated cotton is sometimes squeezed out by hydraulic pressure, and the waste acid runs through iron gutters to a huge tank, into which a stream of mixed acid flows at the same time. The flow is so regulated with regard to the duration of each operation that the composition of the acid mixture remains fairly constant and gives a fairly uniform product. The nitrocellulose for artificial silk is not fully dried, but from 12 to 30 per cent. of water is allowed to remain in it Some patentees claim special advantages, and even a special chemical reaction for variations of 5 per cent. of water either way

The further stages in the manufacture of artificial silk, namely, solution, filtration, spinning, denitration, conditioning, etc., have practically no bearing on explosives, and will not be further dwelt upon.

For the sake of completeness mention must be made of the proposed use of explosives for motive power. I well remember having shown me at Vienna, in 1878, an engine to be worked by small charges of dynamite. In order to show the absence of danger, the inventor had made the model entirely of wood. Again, quite recently my advice was sought regarding the application of smokeless powder to flying machines. Descending to actual

practice, Shaw's pile drivers, worked by gunpowder, are historical, and the riveting machine of Bender,[1] using smokeless powder charges, certainly worked, though I do not know whether it was a commercial success. Several patents referring to motors and compressors driven by explosives have been taken out, and one of them quite recently.[2]

An account of progress on explosives would be incomplete without mention of the conditions under which they are manufactured.

Any one who like myself has occasion to see explosives factories in divers states of efficiency can readily picture the state of small works at a time when regulations and inspections did not exist. Large works had too many interests at stake, and therefore always took some precautions, although, of course, not nearly to the same extent as at the present time. The late Colonel Sir Vivian Dering Majendie deserves lasting recognition for having created that most excellent Explosives Act of 1875. Engineer General Philipp Hess, when still captain, with Captain Trauzl, was instrumental in procuring the carriage of dynamite on the Austrian railways, and shortly afterwards he proposed and in 1877 carried through an Explosives Ordinance, which is practically identical with the British Act. The influence of this Act, and perhaps almost to the same extent of the annual reports of the British Inspectors of Explosives on the arrangements and construction of buildings and machinery, the general cleanliness of the operations performed, and the security of workmen against accident can hardly be overrated, and the example set in this country has been followed all over the world.

In arranging buildings due consideration is now paid to the dangers present on account of the nature of the operation and the quantity of materials dealt with. The advent of high explosives has unfortunately made us acquainted with effects of explosions unknown in the old powder days, and in order to counteract these effects the author recently suggested[3] the construction of danger buildings in a special kind of ferro-concrete. The buildings are so designed that pieces of burning *débris* cannot penetrate their roofs, and so bring about their destruction. At the same time the shock of the explosion transmitted from a distance through the

[1] British patent, No. 26801 of 1898

[2] *Id.*, Nos. 961 of 1874, 24742 of 1904, 28376 of 1904, 22125 of 1905.

[3] Explosions and the Building of Explosives Works, "Journal of the Society of Chemical Industry," 1908, No 13

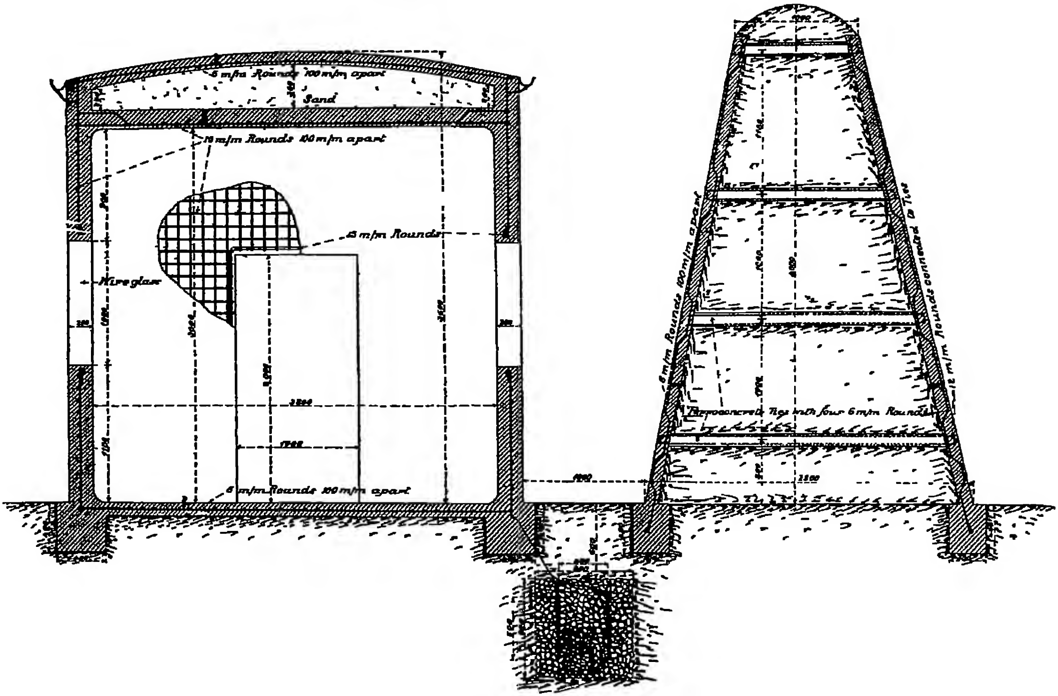

FIG. 11 —EXPLOSIVES WORKSHOP AND MOUND BUILT IN FERRO-CONCRETE

[To face p. 56

ground will not cause the walls to open out. This proposal has been very favourably received by a number of manufacturers, and in several instances has already been adopted. The armouring of such a building forms a Faraday's cage, and renders the whole structure lightning-proof. This is of importance, since the regulations governing the erection of lightning conductors have not increased the safety of buildings to any great extent in spite of lightning-rod conferences and investigations. Magazines which were satisfactorily tested on the very day of a thunderstorm have been blown up, and nothing short of a cage, or at least a complete system of conducting network over and on the buildings, seems to be efficacious.

The construction of the mounds surrounding these buildings seems also to be influenced by a change of ideas. The old plan of erecting stone parapets is again coming into favour. The Author recently observed the effects of an explosion in a dynamite building which was surrounded by solid stone walls The wall on one side only was cracked, the others remained untouched. The communication tunnels were also massively built in stone, and were quite undamaged. This proves the Author's oft-repeated objection to wooden tunnels, which collapse and take fire, to be a valid one. On the other hand the Author has seen sand mounds which appeared to have been lifted up by the shock from an explosion in another building, and to have spread out like putty on falling down again.

Despite all precautions disasters of great magnitude will occur in modern explosives works. This is, no doubt, in the first place, due to the fact that quantities are now-a-days made in such works which were not dreamt of thirty years ago For instance, the works at Modderfontein and Somerset West produce annually over 10,000 tons of dynamite, and several other works run them very close. Such an enormous output requires a very considerable number of buildings, and consequently the chance of damage to life and property is greatly increased. The construction of factories has, on the other hand, proceeded on somewhat orthodox lines, and not always, perhaps, with due regard to subdividing and minimizing risks. Thus the great explosion at Avigliana assumed such enormous proportions because a piece of *débris* from a small building was projected into a magazine several hundred yards away, which latter, exploding in turn, threw *débris* into a number of other buildings. The explosion at Domitz gave rise to precisely similar effects.

Another reason for such catastrophes is the want of appreciation of certain inherent dangers. The Author has always warned manufacturers and users alike that the function of an explosive is to explode, and that although certain compositions are almost insensitive to ordinary impulses, such as blows, friction, etc, yet he never believed that any explosive existed which under favourable conditions and by proper means could not be made to explode It is true that Continental railways carry certain explosives, like ammonium nitrate mixtures, by ordinary goods train, and the Author believes this to be an example which might be quite safely followed by British railway companies in the best interests of the public. There is no danger attached to any of these explosives when in the safe custody of a railway van, and when they do not come into contact with dangerous goods. Even a fire will do no harm, because it is very difficult indeed to set them alight, and almost impossible to keep them alight; so that such goods could soon be moved out of the way. When, however, as was the case at Witten, large quantities of such explosives, and trinitrotoluene and other materials, are massed together or placed close to each other, then nobody can predict the result. It has always been a good rule to keep each risk by itself.

Similarly it will not do to plead ignorance after a number of explosions of picric acid and picrates, potassium chlorate, and the like have been properly investigated and reported upon. Disasters like those at Manchester, Griesheim and St. Helens should not occur again.

Yet another warning to manufacturers may not be out of place. Modern explosives have introduced risks which were unknown to former generations of powder-makers, and even to dynamite manufacturers of long standing. It follows from what has been mentioned concerning the influence of high temperatures on nitrocellulose that special attention must be paid to prevent any accumulation of dirt in places liable to exposure to heat. In the French powder factory at Saint-Médard the explosion which occurred in 1891 could be clearly traced to gun-cotton dust lodging in the joints and cracks of a wooden workshop[1] Do factories even now take every precaution to prevent the accumulation of dirt of this kind? The Author has reason to doubt it, and a clean-up at a factory which he witnessed a short time ago was quite an eye-opener. He can only warn those concerned that every building where explosive dust can be produced, and every appliance and

[1] "Mémorial des Poudres et Salpêtres," 1894, p. 7.

utensil therein, should be periodically and thoroughly cleansed and overhauled More especially is this necessary in the case of drying-, corning-, and sifting-rooms, and the trays, sieves, etc, therein, whose construction should also be such as to prevent any lodgment of explosive dust as far as possible Imagine a drying-tray, covered underneath with canvas, on which gun-cotton or powder is dried all the year round, and ask yourselves what the chemical stability of the dust may be after a year's exposure to a temperature of 40° C. (some factories dry at 50° C), and whether a material dried on such a tray is fairly treated I hope this warning will cause a good many buildings and appliances to be overhauled to their great advantage.

The last twenty years have also witnessed the enormous developments made by electricity When, in 1885, the Author conceived the bold plan of installing electric light in dynamite works on the lake of Lucerne, there was only a shunt-wound dynamo to be had, and it was driven by a water-wheel 10 metres in diameter The fittings had all to be specially made, as there were none on the market. His bold venture was duly punished by a thunderstorm, which flooded the mill-race to such an extent that the water-wheel attained a speed fatal to the dynamo. The year after the Author installed compound-wound dynamos at an Italian factory, but lightning struck the bare overhead wires Happily the large number of lamps burning at the time acted as lightning-arresters. In 1900 the Author's Swiss dynamo was exhibited in the historical section of the Crystal Palace Exhibition; and now, after Waltham Abbey led the way, an explosives factory seems inconceivable without electric light, small motors near buildings or on machines, whilst even the operation of drying sensitive compositions is performed by electric resistances serving as a perfectly adjustable source of heat.

Modern explosives have, on the other hand, introduced electrical dangers themselves. In the first Lecture the possibility of firing a press charge of black powder by static electricity collecting between ebonite press-plates was mentioned. Nitrocellulose is electrified by the current of warm air passing over it when drying, and the necessary earthing arrangements were first proposed by Mr Walter F. Reid, and in many cases especially designed by the Author Mixing machines for blasting gelatine and smokeless powder, especially those provided with reversing gear and belts running in opposite directions, have been known to give long sparks unless properly earthed This was remedied at Waltham

Abbey by saturating the belts with glycerine. The powder itsel during manipulation will generate electricity. A workman a Ardeer, wearing india-rubber-soled shoes on a lead-covered floo was attending to a reeling machine, allowing the strands of powde to pass through his right hand In order to joint a strand, h dipped a finger of his left hand into a bowl of acetone, when h felt a shock, and a spark passed from his finger into the aceton On experimenting, it was found that this result was alway obtained under these conditions, but that when a brass nail wa inserted through the sole, earthing the man through the leade floor, nothing happened.[1] Since that time shoes with coppe rivets were introduced both at Ardeer and at Waltham Abbey Ether vapour given off from smokeless powder and mixed with ai can be fired with a very small spark, and special care should b taken in preventing its formation. The Author recently investi gated a serious accident of this kind. Some smokeless flak powder was being sifted in a reel covered with brass wire gauze The charge was on the point of being finished, the bulk of th powder had been removed, and only a few kilogrammes remaine in three wooden barrels under the outlets of the reel. Suddenly a crackling noise was heard, and the whole of the powder caught fire. One of the two men at work in the building survived, an there can be no doubt from his statement that the two were wel away from the powder, and that this was not touched by anybody The Author could only come to the conclusion that electricity wa generated in the reel, forming a spark when the charge was finished and that the ether vapour, mixed with fine powder dust, ignited.

The manufacture of high explosives seems a simple operation even to experienced chemists, and the danger attending the process appears to be the only difficulty. As a matter of fact, it bristles with difficulties. A good many have already been mentioned, and a few additional and special points are worthy of note.

Glycerine is a uniform, easily-purified substance, and its nitric ester, nitroglycerine, although sensitive to a blow, especially when frozen, is a chemically stable explosive, tame and harnessed for the service of man. Most nitro-compounds of the aromatic series have very great chemical stability. A good many of them may perhaps possess toxic properties, but can otherwise be handled with perfect immunity There are two notable exceptions, however, amongst the explosives at present in use: picric acid and nitrocellulose.

Picric acid is a treacherous substance It is very powerful, but

[1] Report of H.M. Inspectors of Explosives for 1901, p. 37.

that is its only recommendation. Those who use it may be asphyxiated by the fumes of a prematurely exploding shot; those who are fired at sometimes rejoice when it fails to explode. It requires special mixtures to avoid melting at high temperatures, and it attacks its metal container, forming a dangerous picrate. As an ingredient of other explosives it is useless, since on account of its acid properties it reacts upon the other ingredients. Moreover, it is capable of displacing other acids such as nitric acid in nitrates, a disagreeable property which some patentees have found out to their cost. With Montaigne, "I hope that we shall one day give up its use."

A more inconvenient material still is nitrocotton. As already stated cotton is one of the most complex substances known, and for some unexplained reason we have been in the habit of using it after an ill-treatment following upon an undesirable state of cleanliness. At the best, however, we have an almost uncontrollable substance in nitrocotton. It is in such a loose state of equilibrium that the slightest reaction will upset its balance. No wonder that when nitrocellulose is mixed with another explosive like nitroglycerine to form smokeless powder it becomes less reliable, and acts detrimentally on the nitroglycerine. This is accentuated still more in the presence of another disturbing factor such as heat or an alkali. I have seen cases of gelatine dynamite literally dripping with nitroglycerine because a small quantity of ammonium carbonate, added as a so-called "stabilizer," caused the decomposition of some of the nitrocellulose, and this went on to such an extent that the absorbing power of the nitrocotton was destroyed. It is a fact that any alkali, however weak, will gradually saponify the nitrocellulose, and although dangerous decomposition would rarely set in, a bad heat test may result and cause the nitrocellulose to be destroyed by the authorities Chalk in water is no exception to this action

The case is very much aggravated by the action of heat. It is well known that properly purified gun-cotton has been stored in all climates without giving rise to alarming decomposition, even when the temperature was above the normal. Nitroglycerine and nitrocellulose, both of which will by themselves give a potassium iodide heat test of, say, 20 minutes, may, however, when mixed, not stand more than 10 minutes. It is a convenient excuse to say that this is due to an alteration of the physical state, but no proofs have been given for such an assertion, and I should be curious to hear of them.

The amount of nitrous acid required to colour the test-paper is

so small (according to Will[1] it is only 4×10^{-5} milligrams, equivalent to 0·0000016 per cent., or about 1 in 60 millions for a sample of 2·5 grammes) that whatever its physical state, there would always be enough material exposed on the surface to give off this quantity of gas in regulation time if the explosive were of a low order of stability. There is much more justification for supposing that a chemical reaction goes on between the nitroglycerine and nitrocellulose at the elevated temperature of the heat test (82° C.), the nitrocellulose being first decomposed, and the nitrous gases developed reacting on the nitroglycerine and thus accelerating the decomposition.

We next come to the treatment a powder undergoes during manufacture. Whether passed under steam-heated or high-pressure rolls, whether kneaded for hours in a mixing-machine, squeezed from a die with an unnecessary amount of pressure and friction, due to a defect in or bad construction of the die, whether it be dried for weeks and months at temperatures far above the normal, everything tends to destroy the equilibrium of the nitrocellulose. Years ago the Author showed that there is a critical point for mixtures such as blasting gelatine or smokeless powders at or about 45° C., yet during manufacture this temperature is frequently approached and sometimes exceeded.

In some countries the heat test is still carried out at a temperature of 65° C., and if the explosive stands it for, say, 30 minutes, the result is considered satisfactory. Yet how often have we seen this temperature attained during manufacturing operations, and maintained for hours! Is this reasonable?

At a certain powder factory the Author found that the heat tests varied from day to day, and that the ballistic tests were quite puzzling. Investigation showed that the temperature in the drying house was badly controlled, especially at night, and intermittently rose to 50° and 60° C. A simple arrangement rendering it impossible to introduce more heat than that required to maintain the desired temperature made all the difference. Incidentally it may be mentioned that the Author installed an electric alarm thermometer once only, and soon discarded it as it went off too frequently and unnecessarily.

We will assume now that we have taken every precaution to manufacture an explosive, which as regards purity of its ingredients,

[1] Dr. W. Will, "Untersuchungen uber die Stabilitat von Nitrocellulose 2. Mitteilung. Der Grenzzustand der Nitrocellulose in quantitativer Beziehung." Neubabelsberg, 1902, p. 28.

nd as regards care in its preparation, leaves nothing to be desired, nd is therefore perfect in these respects. We now, as behoves areful manufacturers dreading a refusal of deliveries, wish to test mongst other things its stability, and we turn to the purchasing ilitary authorities or the inspecting authorities for guidance. We rere told everywhere until about ten years ago, and are still told so n this country, that the explosive must be heated to a tempera-ure varying from 65° to 82° C without developing sufficient nitrous cid fumes within, say, 10 minutes to colour potassium iodide paper. 'he vagaries of this test are very amusing. Eleven years ago[1] the uthor was the first to show how it could be masked and falsified, nd all that has been done in this country was to carefully regulate he preparation of the sample to be tested, so as to remove all dis-urbing factors. This cannot of course be always assured. For nstance, in smokeless powders, grinding and subsequent drying rill not remove all the solvent, more especially from hard dried owder. With acetone as solvent the heat test will not be a true neasure of the stability, but simply a proof that the sample in uestion is equal to a standard sample, whose purity, together with he amount of acetone left in it, allows a reaction to appear on the est-paper within a certain time. In other words the sample exam-ned is not more masked than the standard sample. These two actors may, however, be quite different in the two samples. The otassium iodide paper itself is an uncertain factor. Great precau-ions must be taken in its preparation, while the thickness of the aper is such a disturbing factor that the papers from one official ource give a test nearly double those from another. In one case ests had to be made on a large number of samples, and four lifferent papers prepared by chemists in authority were used. It vas, however, found quite impossible to obtain uniform results.

Various other tests on similar lines have been proposed to eplace the potassium iodide test, but it would serve no useful urpose to describe them here Some are free from the defects of he iodide test, but not one of them is a true test of stability. 'he potassium iodide, or the diphenylamine test, if always carried ut under identical conditions, are good enough as a rough check n the manufacture to judge whether an explosive material has een sufficiently freed from adhering impurities. They do not, owever, show whether the material itself is so constituted as to emain stable. This is, perhaps, of small importance in the case

[1] The Chemical Stability of Nitro-compound Explosives, "Journal of the ociety of Chemical Industry," April 30, 1897.

of nitroglycerine or an aromatic nitro-compound with their relatively simple structure, but it is all important for nitrocellulose, where the heat test in the opinion of most experts is of little value as a criterion of the finished article. Hess said, in 1879, already,[1] that an explosive may resist well and the impurities may be present in such small quantities as to be insufficient to initiate decomposition of the chief constituents. In this case mere traces would be the only sign of decomposition. After their appearance further spread of the decomposition is either not noticeable for a very long time, or it does not take place at all under the conditions of the experiment. He found this to be the case with several kinds of gun-cotton. In order to judge of stability, the critical point at which an explosive breaks down must be found, and it is also necessary to determine whether decomposition proceeds regularly or at a dangerous and increasing rate when this point is passed. A number of tests have been proposed to fulfil these conditions. They are all based on the principle that a small quantity of explosive is heated to a temperature, which causes decomposition comparatively quickly, yet gives sufficient time to differentiate results. In France this temperature was 110° C, but all the modern so-called "destruction" tests are made between 130° and 135° C.

It was again Hess[1] who, as early as 1879, tried to find the time within which decomposition took place, by passing a current of air over the heated explosive and absorbing the gases by means of a potassium iodide solution. His test was carried out at a low temperature and lasted more than a day. He also indicated a manometric test, measuring the gas pressure developed in unit time. Will made the whole question the subject of most interesting studies, and devised a method by which the rate of decomposition could be accurately and quantitatively ascertained at short intervals of time.[2] Bergmann and Junck proposed another test, in which the gas liberated in a certain time was absorbed in water and its quantity afterwards determined.[3] Obermüller, on the other hand, measures the pressure exerted at constant volume.[4]

[1] "Mitteilungen uber Gegenstande des Artillerie und Geniewesens," 1879, p. 345.

[2] Dr W. Will, "Untersuchungen uber die Stabilitat von Nitrocellulose. Erste Mitteilung. Beurteilung der Haltbarkeit von Nitrocellulose," Neubabelsberg, 1900.

[3] "Zeitschrift fur angewandte Chemie," 1904, p. 982.

[4] "Mitteilungen aus dem Berliner Bezirksverein des Vereins deutscher, Chemiker," 1904, vol. 2.

All these tests require a considerable amount of time and constant supervision by a chemist. Having had occasion to make comparative tests with all the methods proposed, partly in conjunction with Mr. Wm. Macnab, and partly with Mr. G W. Macdonald, the Author has come to the conclusion, that a rapid and reliable method is to heat the explosive in long glass tubes immersed in a bath of amyl alcohol provided with a reflux condenser, and to note the time that elapses before a distinct coloration in the tube is observed. He found that this method compared very favourably with all others. Professor Will [1] has shown, in 1902, that on heating nitrocellulose at a temperature of 135° C. the quantity of gases given off is proportional to the quantity of nitrogen lost. In discussing this result [2] the Author proposed a simple test, whereby with the aid of a sensitive lever balance or some other means the loss in weight of a sample could be watched and determined. This test is now under consideration, and the results are so far very encouraging.

Now let us consider the manner in which the question of stability has been dealt with in practice. The Austrian Ordinance of July 2, 1877, provided in paragraph 51 that the temperature in the magazines indicated by a thermometer constantly kept therein must not exceed 35° C. Of course such a temperature has not been constantly maintained by anybody, unless for some reason it was desirable to dry the explosive at the same time. As I frequently mentioned the duration of the heat test is practically halved by a rise in temperature of 5° C., and Will has confirmed this by proving that the volume of gases evolved is doubled at the same time. This is, however, only correct for temperatures above 45° C., the critical point for nitrocellulose. Below 40° C. the durability of an explosive properly prepared with it increases exceedingly rapidly, and it may be safely assumed that under 20° C. its stability is permanently assured.

This contention has been proved in practice. The author does not know of a single authenticated instance of decomposition in an explosive magazine where the temperature has been kept within the permissible limit.

This simple precaution was, however, neglected in a good many instances by both naval and military authorities. It was and still is the practice in men-of-war to arrange the ammunition stores

[1] Dr. W. Will, "Untersuchungen über die Stabilitat der Nitrocellulose. Der Grenzzustand der Nitrocellulose in quantitativer Beziehung," Neubabelsberg, 1902.

[2] "Chemische Zeitschrift," 1902, p. 371.

and powder magazines in close proximity to boilers and engines, frequently without any ventilation, whilst at times explosives of all kinds are stored together Fourteen years ago[1] the Author discussed this arrangement, and drew attention to the dangers arising therefrom. A dozen explosions on men-of-war and a disaster like that on the *Jena* occurred before an alarm was raised, and now all navies are hurriedly installing refrigerating apparatus. This is all very well as far as it goes if the machinery does not break down at the critical moment, but cannot designers of warships find another place for ammunition? Why go to the length of all sorts of precautions when it should not be impossible to remove the cause of deterioration altogether?

This misplacing of ammunition stores is only slightly mitigated by the fact that twenty years ago the manufacture of smokeless powders had only just begun, and nobody knew much about them. Worse than this, however, was the action of many Governments in at once erecting their own powder works, without any experience in the manufacture of nitro-compounds to go upon, and relying entirely on what private manufacturers cared to show them, and on what they themselves could find out by experiments Some of their powders made fifteen and twenty years ago are still in service, and are now the objects of suspicion.

It is, nevertheless, not fair to throw the whole of the blame on the explosive charge. How would the priming and detonating compositions used in gun charges and shells behave under unfavourable circumstances? Fulminate of mercury, potassium chlorate, sulphur, antimonypentasulphide, picric acid, and other chemicals are contained in such compositions, and it is open to question whether proper tests are always carried out as regards their purity and stability under all conditions. True, Hess has shown[2] that fulminate of mercury can be rendered quite harmless by prolonged heating, but most nitro-compounds explode when suddenly heated, and how the products of decomposition by slow heating would react upon the other ingredients remains to be investigated.

Fearing the lack of stability in smokeless powders, which in the early days of their manufacture was not without justification, inventors began to look around for so-called "stabilizers," that is to say, additional ingredients, which would neutralize the nitrous acid liberated on decomposition. Dr. Dupré's experiments had

[1] "Journal of the Society of Chemical Industry," 1904, p. 583.
[2] British patents, No 3238 of 1902 and No. 13845 of 1902.

shown that the addition of an alkali was undesirable,[1] and the Author's view [2] was often cited with approbation that in a properly prepared explosive a neutralizing agent is unnecessary and may ultimately prove harmful. Other agents were therefore thought of which would have no action on the nitric esters. Some people thought that if a little ether-alcohol was left in the powder, it would act as a stabilizer, and in order to prevent the rapid escape of the solvent on storage, a little amyl alcohol was added, thus slightly raising the boiling-point of the solvent[3] As a matter of fact this would merely constitute absorption of the nitrous vapours, but would not prevent their being given off again on heating.

A better plan is the addition of "stabilizers," which would form stable compounds with the nitrous acid; for instance, aniline, which the Nobel factory at Avigliana used in their gun-cotton twenty-four years ago, and which both they and the Italian Government employ for Service Ballistite Diphenylamine and, it is said, vaseline would act in a similar way. There is a great difference between such real "stabilizers" and alkaline "neutralizers." In the latter case nitrites are formed, which constantly release nitrous acid on prolonged heating, and reabsorb it again, thus acting as catalysers. The stable compounds formed from stabilizers, like amidoazobenzol, and other aromatic nitro-compounds, retain the nitrous acid, and thus transform the reaction into a slow and regular one, which keeps the powder in good condition as long as there is any stabilizer left. The length of time a powder remains in good condition will therefore only depend on the proper constitution and manufacture of the powder. Possibly more strenuous advocates of the iodide heat test will again complain of a vitiation of their test, but there are apparently good and valid reasons for considering such additions useful, and they will have to be considered as legitimate ingredients of the powder. This has already been done in some instances.

Stabilizers, like diphenylamine and aniline, will also reveal their presence, as soon as the powder goes wrong, since the compounds formed with them by the action of nitrous acid show as spots or stripes of peculiar colours, varying either in shade or intensity as decomposition progresses. Since the French Commissioners on

1 "Report of Her Majesty's Inspectors of Explosives for 1887," p. 21

2 "The Manufacture of Explosives," London, 1904, vol. ii, p. 72.

3 Chambre des Députés, "Rapport sur les causes de la catastrophe de l'Jena," Paris, 1907.

the *Jena* accident emphasized this fact, already known in Germany and Italy, everybody speaks of "révélateurs," the addition of an indicator, as being a panacea. As a matter of fact the Author considers it only a needlessly alarming arrangement, like an alarum thermometer, and unnecessary with a good powder stored under proper conditions, but which would cause commanders of warships to nervously watch their stores after the faintest indication, without giving them any remedy in mid-ocean. This is very much like the piece of litmus paper which, according to the Austrian ordinance of 1877, has, I believe, still to be kept in every case of dynamite, although with the present-day perfection in manufacture nobody wants it. The whole idea is not new, having been patented by Nicholson and Price in 1871.[1]

Whilst the Author believes such additions of real stabilizers to be an important improvement, he cannot consider the question of smokeless powders to be quite satisfactorily solved thereby.

What we must aim at is an explosive which is durable and stable under all ordinary conditions of use, and even under some extraordinary ones, just as in the case of the old black powder. In the Author's opinion, and his view is shared by very eminent colleagues, there can be no doubt that nitrocotton (and for the matter of that any other nitrocellulose) is not a suitable ingredient for a Service powder. Having built or reconstructed a number of works, and seen quite half of all those in Europe, he ventures to speak with some authority. Let us again recapitulate its defects. Made from a material which is most complex and liable to form unstable compounds, we elect to use it in a form which can neither be clean, nor of uniform growth, nor even of constant composition. The conditions of manufacture are such that in the absence of very special precautions the nitrocotton retains unstable compounds, and is liable to decompose. Under the influence of heat, of certain additions or ingredients, of unsuitable treatment or friction, the nitrocotton may decompose and react in a progressive manner upon the other ingredients. It requires a solvent in order to be brought into a physical state which will permit the rate of burning of the powder to be regulated. Such solvent, if volatile, requires prolonged heating to drive it off as completely as possible. This heating helps to shorten the life of the powder, and any solvent remaining behind affects its ballistic properties. Nitrocellulose is not a uniform compound by any means, and it is almost impossible to make sure that every batch shall have the same composition and

[1] British Patent, No. 2430 of September 15, 1871.

effect. The latter by no means depends on the percentage of nitrogen being the same, though this condition may be fulfilled by suitable blending. For instance, a mixture of soluble and insoluble nitrocellulose would not have the same effect as a nitrocellulose prepared direct, although each may contain the same percentage of nitrogen.

The question will naturally be asked, What will be the powder of the future? If we may venture a prophecy, the future belongs to a stable nitro-compound of the aromatic series, perhaps in conjunction with nitroglycerine. Such nitro-compounds have already been proposed, and sooner or later one will be found that meets all requirements. Although every Service will be reluctant to make a change, yet having learnt to appreciate the value of scientific research, some Government will be sure to make a bold plunge, when all others will soon follow suit.

APPENDIX I

Translation. No. 21/208.

DESCRIPTION OF THE MANUFACTURE OF A CHEMICAL RIFLE AND BLASTING POWDER, FOR THE PATENTING OF WHICH I HAVE APPLIED UNDER TO-DAY'S DATE.

For the manufacture of this powder leaf-wood should be used, alder-wood, cut down during the winter months, being the most suitable All other varieties of wood are not so suitable The description of the manufacture of this powder may be divided into two chief stages, viz.—

I. Mechanical Treatment of the Wood,

and II. Subsequent Chemical Treatment.

I. Mechanical Treatment of the Wood.

The wood is divided by means of suitable instruments into bodies so small that after they have been dried the size of the grains is the same as that of the well-known Black Rifle and Blasting Powder

II. The Chemical Treatment of the Particles of Wood thus obtained, which for the Purpose of Description may be Divided into Five Stages, viz —

(*a*) *Removal of acids and readily soluble materials.*
(*b*) *Removal of substances containing albumen.*
(*c*) *Removal of colouring matter*
(*d*) *Treatment with nitric acid.*
(*e*) *Impregnation with salts, containing oxygen and nitrogen.*

(*a*) *Removal of acids and readily soluble material.*

After placing sufficient water in a boiler (preferably of copper) to permit of 100 lbs. of grains of wood mentioned in I. to float therein, 3 lbs. of sodium carbonate are dissolved in it, next the above mentioned 100 lbs of wood grains are added and the whole boiled for 3 to 4 hours,

at the end of which period the liquid which has become dirty is poured off and replaced by fresh, containing the same proportion of sodium carbonate, after again boiling the grains for 3 to 4 hours they are exposed for 24 hours to cold running water

(b) *Removal of substances containing albumen.*

After the grains have been subjected to the treatment described under (a) and have subsequently been dried, place them in a vessel with a sieve-like bottom, and pass so much steam through them for 15 minutes that, with the aid of stirring, the substances containing albumen are detached and carried away, after this the grains are exposed for about 24 hours to cold running water and then dried.

(c) *Removal of colouring matter.*

After the grains have been subjected to the treatment described under (b) the removal of the colouring matter is effected by the use of a solution of bleaching powder or chlorine gas, the choice of one or the other depending on purely economic considerations

If bleaching powder is to be used 15 lbs of this are dissolved in 260 lbs of water in a vessel, which must afterwards be closed up air-tight. The grains are placed in a vessel, and enough of the above solution is poured on them to cover them, in doing which the fact that the grains absorb moisture must be considered

After the grains have been stirred in the liquid for about 2 hours they are washed in fresh running water, then placed in clean water and heated to boiling, after which they are exposed for 24 hours to fresh running water, and then dried in moderately warm air.

If chlorine gas is preferred this is passed through a vessel containing the grains and provided with a sieve-like bottom, until they are discoloured, whereupon they are washed in running water and dried in moderately warm air.

(d) *Treatment of the grains with nitric acid after they have undergone the treatment described in* (c)

Take 40 parts by weight of concentrated nitric acid, which must have a specific gravity of at least 148°, mix them with 100 parts by weight of sulphuric acid, which must have a specific gravity of at least 184°, stir the two together for two hours and put in a cool place. Next put 100 parts by weight of this mixture into an iron vessel, which one must constantly keep cool by placing it in cold running water, or by other means, and gradually mix into it 6 parts by weight of the grains, stirring meanwhile continuously. After stirring the grains for two hours in the acids, dry them by means of a centrifugal machine, or

similar apparatus, and expose them for two days to cold running water, after which they are brought to the boiling point in a weak solution of sodium carbonate, again exposed to cold running water for 24 hours and then dried.

(*e*) *Impregnation of the grains with salts that contain oxygen and nitrogen, after having undergone the treatment described in* (*d*).

For this saltpetre, together with barium nitrate, or else saltpetre alone are used, economic considerations determining this. If saltpetre and barium nitrate are taken 22·5 parts by weight of saltpetre and 7·5 parts by weight of barium nitrate are dissolved in 220 parts by weight of water at 112° F, whereupon 100 parts by weight of the grains are added and stirred for 10 to 15 minutes If saltpetre alone is preferred 26 parts by weight of it are dissolved in 220 parts by weight of water at 67 5° F, 100 parts by weight of the grains added and stirred round for 10 to 15 minutes In both cases the grains are subsequently dried at a temperature of 110 to 112° F

The material manufactured in the manner described so far is a perfectly serviceable powder, yellow in colour, and far superior to the universally used black gunpowder, compared with its weight it is, however, so voluminous that it cannot be used in barrels having a very small space for the charge, and could not be used economically for blasting purposes, as in the latter case it requires too large—*i. e.* too costly—bore-holes, for which reason I proceed in the treatment of this powder in accordance with the following description of its manufacture in order to render it applicable for all purposes

Treatment of the chemical gunpowder with alcohol and ether.

Take 5 volumes of pure ether and one volume of pure absolute alcohol and put them in an air-tight vessel Next place the previously made yellow chemical rifle-powder into a vessel which can also be closed up air-tight, and allow as much of the mixture of ether and alcohol to enter as is required to completely saturate the powder. Leave the powder in this bath between 3 and 30 minutes, according to the purpose intended, and according to whether the powder consist of large or fine grains

The action of this bath causes the cellulose to dissolve, *i. e.* in the present case a solution of the wood fibre in the grains of powder, and solution takes place sooner or later according to the duration of the bath, and according to whether the powder consists of large or of fine grains.

If it is desired to keep the powder, which has been poured into the bath, in its granular form, the bath must not be of such duration that the wood fibres in the grains are completely dissolved, but merely

sufficient to cause this destruction to take place in the outer walls of the single powder grains only, and further, when the liquid in the bath has been allowed to run off and the grains have been spread out, all pressure on them must be avoided when they are in superimposed layers, as these would immediately form a solid mass; on the contrary, these grains must be continuously stirred, and at the same time so much dry and untreated powder grains must be gradually sprinkled in between them until all tendency to adhere to each other has ceased. This addition of untreated grains may be dispensed with if by another arrangement, *e.g* by leading in air which keeps the mass of treated grains in motion, their sticking together is prevented. After this the powder is first dried for 12 hours at 21° R, and then for 24 hours at 40° R

If it is desired to make the powder in solid pieces the duration of the bath is, within a limit of 30 minutes, made such that that degree of solution of the wood fibres has taken place as is necessary for the required purpose, in every case the grain powder poured into the bath must have been converted into a mush, which is subsequently dried for 12 hours at 15 to 24° R. (according to the diameter of the mass), and thus converted into a dough-like pliable substance, the latter can be brought into any desired form by using moulds and presses, these forms being then subjected first to a temperature of 24° R. for 24 hours, and then to one of 40° R for another 24 hours, to dry them The grains so dried have the consistency of wood.

The speed of evolution of the gases from this powder is produced, or rather, depends on the more or less complete solution of the wood fibres in the grains of powder, and on the greater or lesser amount of pressure given to the first mush, and subsequently dough-like substance so obtained: rules based on experience enable the manufacturer to regulate this mathematically exactly. I would add that the words "pure ether" and "absolute alcohol" mean that the former must be chemically pure, and the latter must have at least 96° Tralles.

By the description of the manufacture of chemical gun- and blasting powder hereby completed, I have proved the formation of three varieties of this powder.

A. The original variety in the form of grains, yellow in colour, for the manufacture of which no ether and alcohol has been used.

B. The variety obtained in the shape of grains, after using ether and alcohol.

C. The variety obtained as solid bodies of any desired shape after using ether and alcohol, *B and C are brown in colour.* It only remains for me to discourse on the properties of this chemical powder compared to those of the hitherto universally used black powder, and in speaking of the peculiarities of the three varieties, I shall, for the sake of

brevity, use the letters A, B, and C, already mentioned, to denote them.

The following are the properties of A—

1. Its vapour on firing is of such a transparent nature that with rapid or heavy fire, at the same time and in the same place, the eye of the firer is never prevented from seeing the target.

2. Its report is much less than that of black powder, which is of importance in ships and in casemates.

3. It leaves a very small amount of dry ash-like residue, which is always cleared away by the next shot, so that the interior of the rifle or gun barrel is always metallically clean, and the shot is therefore able to follow the rifling

4. Only half the weight of that of black powder is required to send the projectile one-third as far again, with a quarter greater muzzle velocity.

5. With this powder the trajectory of the shot is half as straight again, which will be of advantage in the making of rifles, *e.g* in the army rifle the hinged sight would be discarded.

6. Its effect is always uniform, since the gas-producing elements do not emanate from a mechanical mixture of solid bodies, but are presented to the single grains as a liquid for absorption.

7. There is no danger in manufacture.

8. There is no danger in storage, as it only explodes when imprisoned, as in the rifle barrel, or in the rock to be blasted, but otherwise merely burns with a bright flame.

9. It is free from danger in transport, and even absolutely without danger should fire accidentally break out, provided that it is packed in sacks or in a wet state, for—

10. Moisture does not harm the powder, and if it has been saturated with water for months, it only requires to be dried to possess its whole former effect The two varieties of powder B and C possess the following advantages in addition to those just enumerated—

1 The result of the action of the ether and alcohol bath is the solution of the wood fibres in collodion, whereby the volume is considerably reduced in relation to the weight, and that the powder substance, whether in the form of bodies, as in B, or as a solid mass, as in C,

2. possesses no affinity whatever for moisture, since both in the case of the grains and in the solid form this is entirely closed up by the collodion, and this to such an extent that immersion for any length of time in running water is without action thereon.

3. In addition to being now fit for use in all kinds of rifles and guns after the diminution in volume, it is the most suitable material for all blasting purposes, since it occludes so great a force in a small

body as no other known substance, and this is so important because four-fifths of the cost of blasting goes to the bore-holes, and only one-fifth to the price of the blasting agent.

Samples of the three kinds—A, B, C—of the powder are enclosed.

(*Signed*) FRIEDRICH VOLKMANN,
Gentleman,
13, FLORIANIGASSE, VIENNA

VIENNA, *November* 8, 1870
Granted, June 11, 1871.

APPENDIX II

Translation No. 21/257.

DESCRIPTION OF THE MANUFACTURE OF A NEW RIFLE AND BLASTING POWDER, FOR THE PATENTING OF WHICH I HAVE APPLIED UNDER TO-DAY'S DATE.

FOR this purpose leaf-wood should be used, alder-wood being the most suitable.

I. MECHANICAL TREATMENT OF THE WOOD.

The wood is shaped by means of suitable cutting and sawing instruments into bodies such that, after drying, the size of the grains is as nearly as possible that of the well-known black gunpowder.

II. THE CHEMICAL TREATMENT OF THE PARTICLES OF WOOD OBTAINED IN ACCORDANCE WITH PAR. I., WHICH MAY BE DIVIDED INTO SIX STAGES, VIZ.—

(a) *Removal of acids and readily soluble materials.*
(b) *Removal of albuminous matter.*
(c) *Removal of colouring matter.*
(d) *Treatment with nitric acid.*
(e) *Impregnation with salts, containing oxygen and nitrogen.*
(f) *Treatment with ether and alcohol.*

(a) *Removal of acids and readily soluble material.*

Three lbs. of sodium carbonate are dissolved in a boiler (preferably of copper) in a quantity of water sufficient to permit 100 lbs. of grains of wood mentioned in I. to float therein; next, 100 lbs. of wood grains are added and boiled for 3 to 4 hours, at the end of which period the liquid which has become dirty is poured off and fresh liquid added, boiling proceeds again for 3 to 4 hours, after which the grains are exposed for about 24 hours to cold running water.

(b) *Removal of albuminous matter.*

After the grains have been subjected to the treatment described under (a) and have subsequently been dried, place them in a vessel

with a sieve-like bottom, and pass so much steam through them for 15 minutes that, with the aid of stirring, the albuminous matter is detached and carried away; after this the grains are exposed for about 24 hours to cold running water and then dried.

(c) *Removal of colouring matter.*

After the grains have been subjected to the treatment described under (b) the removal of the colouring matter is effected by the use of a solution of bleaching powder, or also chlorine gas.

If bleaching powder is preferred, 15 lbs. of this are dissolved in 260 lbs of water in an air-tight, closed vessel.

The grains are placed in a vessel and enough of the solution is poured on them to cover them with liquid after they are saturated. The vessel having been covered as tightly as possible the grains are stirred in the liquid for about 2 hours, after which they are washed in fresh clean water. The grains are next treated in clean water to the boiling point, after which they are exposed for 24 hours to cold running water, and then dried in moderately warm air

If chlorine gas is preferred, this is passed through a vessel containing the grains and provided with a sieve-like bottom for a period sufficient to bleach them, whereupon they are washed in cold water and dried in moderately warm air

(d) *Treatment of the grains with nitric acid after they have undergone the treatment described in (c).*

Take 40 parts by weight of concentrated nitric acid, which must have a specific gravity of 148°, and mix this with 100 parts by weight of sulphuric acid, which must have a specific gravity of 184°, stir the two together for two hours and put in a cool place.

Next put 100 parts by weight of this mixture into an iron vessel, which one must endeavour to keep cool by placing it in cold running water, or by other means, and mix into it 6 parts by weight of the grains, stirring meanwhile continuously, take the grains out of the liquid and dry them by means of centrifugal or similar machines. The grains are then exposed for two days to cold running water, after which they are brought to the boiling point in a weak solution of sodium carbonate, again exposed to cold running water for 24 hours and then dried

(e) *Impregnation of the grains with salts that contain oxygen and nitrogen after having undergone the treatment described in (d).*

For this, saltpetre together with barium nitrate, or else saltpetre alone, are used.

If saltpetre and barium nitrate are taken, 22 5 parts by weight

of saltpetre and 7·5 parts by weight of barium nitrate are dissolved in 220 parts by weight of water at 112° F., whereupon 100 parts by weight of the grains are added and stirred for 10 to 15 minutes.

If saltpetre alone is taken 26 parts by weight are dissolved in 220 parts by weight of water at 67 5° F., 100 parts by weight of the grains added and stirred round for 10 to 15 minutes.

In both cases the grains are subsequently dried at a temperature of 110° to 112° F.

(*f*) *Treatment with ether and alcohol*

Take 5 volumes of chemically pure ether and 1 volume of absolute alcohol, which must be at least 96° T., and mix them in an air-tight vessel; next place in another vessel, which must also be closed air-tight, and which is connected to the first vessel by a pipe, sufficient of the grains obtained from (*e*) to make it about three-fourths full, close it and allow so much of the mixture of ether and alcohol to flow through the connecting pipe on to the grains, that after the intended degree of saturation has been obtained they are covered by the liquid. Allow the grains to remain in this bath for 3 to 30 minutes, according to the object desired, the size of the grains being the determining factor. Owing to the wood pulp in the grains being dissolved by the action of the bath, the grains are brought into a pulpy condition, so that, if it is desired to keep the powder in the form of fine grains, the latter must not be exposed to the bath long enough to enable the solution of the woody matter to extend right into the middle of each grain, and further, in this case, after the grains have been taken out of the bath all pressure on them must be avoided, as they would thereby be converted into a solid mass By stirring continually, and best of all, by means of air flowing through from underneath, and at the same time sprinkling dry powder dust over them, it is possible to keep the grains separate. Once the grains have lost their tendency to stick to each other they may be superimposed first of all in thin and then in thicker layers, and they are then dried first of all for 12 hours at 24° R. and then for 24 hours at 40° R.

If it is desired to obtain the powder as a solid body the grains must be exposed to the bath sufficiently long to enable the process of solution to penetrate deep enough for the purpose intended The further solution of the woody matter is carried the less the volume becomes, and the more solid, and the smaller for equal weights do the bodies become that can be obtained on pressing. If in such a case the powder is taken out of the bath it has the appearance of a pulp, which after being dried for 12 hours at a temperature of 24° R will change into a dough, which can be made to take any desired shape by means of moulds and presses. A greater or lesser degree of solution of the

woody matter or of pressure exerts a great influence on the *kind* of effect produced by the powder bodies, so that their rapidity of combustion as rifle powders can be varied indefinitely, and one can impart to them as blasting agents the gentle lifting action desirable for slate quarrying, or also that violence needed for breaking up rock

Having herewith completed the description of the mode of making this my rifle and blasting powder, I would add that small departures, both with regard to the quality and to the quantity of the substances used, and also minor departures in manipulation during manufacture, do not affect the *formation* of the powder in general, but only its *quality*.

The advantages of this powder over the chemical wood powder are that its volume is considerably reduced in relation to its density, whereby it can be used even for rifles having a very small chamber for the charge, and is more economical for blasting purposes, further, that its affinity for moisture is reduced to such an extent that however long it may be immersed in *running* water its efficacy remains unimpaired, and that it possesses all the other properties of the former powder to a greater degree.

(*Signed*) FRIEDRICH VOLKMANN.

VIENNA, *February* 3, 1871
Granted, May 31, 1871.

INDEX

ABEL, Sir Fredk., 2, 25, 38
Abelli, Dr., 29
Absorption towers, inverted, in mines, 51
Acetone, use of, 42
Acid, dilution of waste, 21
— waste, from gun-cotton, 28
Acids, revivification of waste, 33
Alberts, 12
Alkalies, 61
Allendorff, 15
Aluminium explosives, 11
— powders in detonators, 45
Amide powder, 5
Ammonal, 11, 52
Ammonite, 52
Ammonium nitrate, explosion of, 50
— — safety explosives, 46
Ammunition, storage of, in the Navy, 66
Amyl alcohol, 41, 67
Aniline, 41
— as stabilizer, 67
Arkite, 52
Avigliana explosion, 57

Bacon, Roger, 1, 9
Ballistite, 40, 67
— manufacture of, 41, 42
Bavarite, 16
Bobie, 26
Bellite, 52
Bender, 55
Bergmann, 7, 64
Berthollet, 1, 8
Bevan, 2, 26
Bichel, 15, 48, 49
Bichromates, 49
Bickford's safety fuses, 46
Bielefeldt, 45
Black blasting powder, sale and exportation of, 3
— powder, improvements in, 4
— — sifting, 8
— — with sodium nitrate, 4
Blasting gelatine, invention, 2, 22, 23
Blount, Bertram, 6
Bobbinite, 5, 6, 48, 49, 52, 53
Boulanger, General, 2
Bowman, F. H., 27
Brank, 9
Breguets, low tension fuses, 46
Brown, 25
— powder, 3
Brownsdon, Dr., 45
Brugère's powder, 16
Bruley, 31
Bruyn, Lobry de, 50

Cahuecite, 4
Camphor, 41
"Canary birds," 13
Carbonite, 48, 49, 52
— rye flour in, 50
C/89 powder, 41
Celluloid industry, 54
Centrifugals, nitrating, 32
Chapman, 11
"Charge limite" of an explosive, 50
Cheddite, 9
Chlorate mixtures, 8
— powder, 1
Cleanliness in factories, 58, 59
Coal dust, 51
— mines, explosives in, 47
Colliery Steelite, 10
Collodin, 38
Collodion cotton, 22, 23
Commission de Pyroxyle, 37
Concrete, danger buildings in, 56
"Condensed" powders, 45
Cordeaux détonants, 47
Cordite, 41
Cotton, bleaching of, 26
— dead, 27
— fibres in polarized light, *Frontispiece*
— impurities in, 26
— nitration of, 29
— selection, 29
— substitutes for, 29
— unstable nitro-compounds of, 27
— waste, 25
Cross, C. F., 2, 26

Dahmen, Ritter von, 11
Danger buildings in ferro-concrete, 56
— — mounds for, 57
Dead cotton, 27
Delaval, Thomas, 1
Designolle, 1
"Destruction" heat test, 64
Detonating compositions, stability of, 66
— fuses, Hess' phlegmatized, 47
Detonators, 45, 46
— with trinitrotoluene, 15
Diglycerine, 24
Dinitroacetine, 25
Dinitrodichlorhydrine, 25
Dinitrodiglycerine, 24
Dinitroformine, 25
Dinitroglycerine, 23, 24
Dinitromonochlorhydrine, 24
Dinitrotoluene, 23
Diphenylamine, 41
— as stabilizer, 67

Displacement process, 21
— — for nitrocellulose, 34
Domitz explosion, 57
Drying house, 62
Dupré, Dr. A., 7, 8, 66
Duttenhofer, 43
Dynamit Actien Gesellschaft, Hamburg, 20

Ebonite, in presses, 7, 8
E.C powder, 2, 39
Ecrasit, 13
Egg, Durs, 45
Electricity, static, in presses, etc., 59, 60
Electric lighting of explosives factories, 59
Engler, 51
Escales, Dr. Richard, 11, 24
Ether acetic, effect on powder, 37
— alcohol as a stabilizer, 67
— vapours, 60
Explosions in factories, 57
Explosives Act, 1875, 56
— for motive power, 55
— safe, 50
— safety in fire-damp, 48

Fabrik elektrischer Zunder, 46
Fabrikoid, 54
Faversham powder, 52
Ferro-concrete danger buildings, 56
— -silicon, 11
Fibre, use of, in presses, 8
Fire-damp, 51
Flame, cooling agents, 49
— effect of, on safety of explosive, 49
Flameless powder, 43
Flour, rye, 50
Förster, Max von, 33, 39
Fraenkel, 9
Frameries, testing gallery at, 51
Friese-Green, 11
Fulminate of mercury, 45, 46
— — stability of, 66
Fungi in nitrocellulose, 33
Fuses, Bickford's safety, 46
— electric, 46
— friction, 47
— Hess' phlegmatized, 47
— improvements in safety, 5
— safety, 46

Gehre, 16
Gelatine, blasting, 22, 23
Girard, 12, 47
Glycerine for belts, 59
Gœpner, C, 20
Griesheim, Chemische Fabrik, 13
Gun-cotton, 25
— compressing large charges, 35
— dust explosion, 58
— invention, 2
— powder, 2

Gunpowder, invention of, 1
— with coal brasses, 1
— without sulphur, 1
Guttmann, Oscar, 6, 12, 29, 40

Hahn, F., 9
Hall, 51
Haller, Dr. R , 27
Hartig, Dr., 36, 37, 40
Hauff, 13
Häussermann, 2
Heat, action of, 61
— test, 61, 62, 63, 64, 65, 67
— — destruction, 64
— — masked, 63
Hebler, Prof , 39
Hell Gate rocks, blasting of, 10
Hellhoffite, 48
Hellich, Major, 6
Hess, General, 47, 56, 64, 66
Himly, 9
Hirtenberg, magazines exploded, 2
Hoitsema, 35
Hollings, 34
Hough, Arthur, 35
Howard, 45
Hyatt, Brothers, 54
Hydrocellulose, 27
Hyronimus, 45

Immersion apparatus for nitrocellulose, 33

"Jena," explosion on the Battleship, 66, 68
Jenks, 26
Junck, 64

Kast, Dr. H., 24
Kelbetz, 7
Kollner, Dr., 2, 38
Kieselguhr for dynamite, 22
Kinetite, 48
King's Norton Metal Co., 45
Knell, 11
Korda, Désiré, 11

Lauer, General, 47
Lavoisier, 1, 8
Le Blond, 1
Lehmann, 24
Lenck, Baron von, 25, 37
Lenze, Prof., Investigation on Perchlorate, 7
Lewall, 9
Liecke, 23
Liedbeck, Alarik, 17
Lighting, electric, of explosive factories, 59
Lightning, protection of danger buildings against, 57
Litmus paper as indicator, 68
Lundholm, 42

Lyddite, 13
Lyte, 9

Macdonald, G. W., 65
Machinery for black powder, 7
Macnab, Wm., 65
Magnesium, 11
Majendie, Col. Sir V. D., 56
Malenkowicz, 33
"Maltha," 9
Margraf, 48
Martin, 51
Matter, 45
Mayer, black powder, 5
Melinite, 12, 13
— for fuses, 47
Melting points of mixtures, 12
Memmingen, Abraham von, 36
Men-of-war, storage on, 65
Mercury, fulminate of, 45, 46
Mettegang, Dr., 49
Mikolajczak, Dr. Anton, 23
Military powders, requirements of, 43
Mine galleries, watering of, 51
Mining Association of Great Britain, 51
Mixing machines, 59
Monobel powder, 52
Monochlordinitroglycerine, 24
Monochlorhydrine, 24
Mononitrodichlorhydrine, 24
Mononitroglycerine, 23
Montaigne, 36
Mosenthal, Henry de, 2, 21, 22, 31, 44
Moss, 49
Motive power, explosives for, 55
Mounds for danger buildings, 57
Mowbray, 17

Nachscheidung, 20
"Nassbrand" powder, 4
Nathan, Lt.-Col. Sir Frederic L., 18, 19, 21
Nathan, Thomson and Rintoul's process, 19
Nauckhoff, Sigurd, 24
Neumayer, G. A., 4
Neunkirchen, testing station at, 48
"Neutralizers," alkaline, 67
Nicholson, 68
Nitrating centrifugals, 32
Nitrobenzene, 23
Nitrocellulose, 2, 54
— boiling, 31, 32
— critical temperature for storing of, 65
— drying with alcohol, 41
— effect of varying single factors, 30
— electrification of, 59
— granular, 38
— powders, manufacture of, 41
— pulping, 31
— requirements for artificial silk and varnishes, 55
— stability of, 61
Nitro-compound, first, 36
Nitrocotton, 61
Nitrocotton, unsuitability of, for a Service powder, 68
Nitroglycerine, 18, 60
— invention, 2
— unfreezable, 23
— yield of, 19
Nitrohydrocellulose for fuses, 47
Nitrostarch, 35
Nitroxylin (see also Schultze powder), 38
Nobel, Alfred, 2, 17, 22, 23, 25
— — invents Ballistite, 40
— factory, Pozsony, nitrating mixture, 18

Obermüller, 64
Oil, addition to chlorate explosives, 9
Oils in flameless powders, 43
Oleum benedictum, 36
Otto, Prof., 36
Oxalates, 49
Oxycellulose, 27, 28

Panactovic, Dr., 6
Paraffin in nitroglycerine manufacture, 20
Pegamoid, 54
Pendulum ballistic, use of, 50
Perchlorate in saltpetre, 6
Permanganates, 49
Permonite, 10
Pernice & Co., Volkmann's k. k. priv. Collodinfabriks Gesellschaft, 38
Pertite, 13
Petroklastite, 4
Phlegmatized fuse (Hess'), 47
Picrates, 58
Picrate powder, 1
Picric acid, 2, 12, 58, 60
— powder, 15
Picrinit, 13
Pile drivers, 55
Pit gas, 51
Plane sifter, 8
Plastrotyl, 15
Potassium chlorate, 58
— iodide heat test, 63
Poudre B, 2
"Poudre peigne," 42
Price, 68
Priming composition, stability of, 66
Products of combustion, effect on safety of explosive, 49

Rackarock, 10
Railways, transport of explosives on, 56, 58
Reese, Dr. L. F., 20
Refrigerating apparatus in the Navy, 66
Reid, Walter F., 2, 39, 40, 59
"Révélateurs," 68
Rheinisch Westfälische Gesellschaft, 45
Rintoul, 18, 19, 21
Rippite, 52
Riveting machine, 55
Robertson, Dr. R., 31, 32
Roburite, 48, 52

oscoe, Sir Henry, 30
udberg, A. E., 23
udeloff, 15

ıfety blasting powder, 4
— explosives, 2, 46, 47
— — definition of, 48
— — quantity of, consumed in Great Britain, 52, 53
— fuses, Bickford's, 46
alt, common, 49
altpetre, perchlorate in, 6
and, 49
axonite, 52, 53
ayers, 42
chmidt, 48
choeneweg, 49
chonbein, 1, 36
chultze, Captain Eduard, 37, 40
— powder, 2, 29, 38, 47
chulz, Professor, 16
chwarz, Berthold, 36
— Richard, Dr., 54
Schwarzkoppen, von, 37
Securite, 48, 49
Shaw, 55
Shimose powder, 13
Siersch, 49
Silicon and its alloys, 11
Silk, artificial, 33
— — production of, 54
Silver nitride, 45
Simon, 51
Smokeless powder, 2
— — defects of, 44
— — electrification of, 60
— — history of, 36
— — various shapes of, 42
Soaps in flameless powders, 43
Sobrero, Ascanio, 2, 17
Société des Poudres et Dynamites, 23
Sodium bicarbonate, 43
— fluoride in nitroglycerine manufacture, 20
Solenite, 42
Solvent naphtha, 16
Sporting powders, requirements of, 43
Sprengel, Dr. Herrmann, 2, 10, 12
Sprengsalpeter, 4
Stability of nitrocellulose, 65
Stabilizer, 61, 66
Steelite, colliery, 10
Stone dust in mines, 51
Stowite, 52
Street, 9
Sulphuric anhydride, 18
Suter, 55
Swiss powder, 4

Temperature, critical, 62
— — for storing nitrocellulose, 65
— of explosion, 48, 49
Testing stations, 50
Tetralit, 15
Tetranitrodiglycerine, 24
Tetranitromethane, 10
Tetranitromethylamine, 15
Tetranitromethylaniline for detonators, 45
Tetryl, 45
Thermometer, alarm, 62
Thomson, 19
— James Milne, 34
— William, 34
Tiimann, 47
Tolite, 14
Tonite, 47
Tonkin, John, jun , 25
Trauzl, Captain, 56
Trinitrobenzene, 13
Trinitrobenzoic acid, 13
Trinitroresorcine, 13
Trinitrotoluene, 13
— for fuses, 47
— in detonators, 45
Trinol, 14
Triplastit, 15
Trotyl, 14
Tschirner, 9
Turpin, Eugène, 2, 12

Vacuum vessel for lifting nitroglycerine, 22
Varnishes, nitrocellulose for, 54
Vaseline, 41
— as stabilizer, 67
Vender, 25
Vieille, 2, 40
Vignon, 2, 27
Vigorite, 16
Volkmann, Friedrich, 2, 40, 71, 77
— — invents smokeless powder, 38, 39

Walsrode powder, 44
Water cartridge, 47
— for shooting, 36
— jacket, effect of—round charge, 49
— of crystallization for cooling explosives, 49
— spray in mines, 51
Watering of mine galleries, 51
Watteyne, 50
Weiffenbach, 11
Wenghœffer, 11
Westfälisch Anhaltische Gesellschaft, safety fuse, 46
Westfalite, 52
Wetter-Dynammon, 5
Will, 2, 24, 28, 62, 65
Winand, 10
Witten explosion, 58
Wohl, 24
Wohler, 45
Wolff & Co , 15
— Oscar, 39
Wood pulp, 50
Woolwich, testing gallery at, 50

Yonckite, 10